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A Journal of Practical Chemistry

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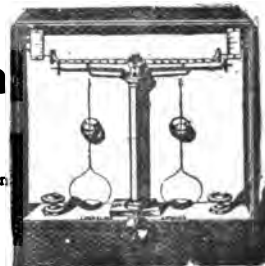


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THE CHEMICAL NEWS.

VOLUME LXXXVI.

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No. 2223.—JULY 4, 1902.

THE COMPOSITION OF COLOSTRUM.

By WALTER F. SUTHERST, Ph.D., A.I.C.

COLOSTRUM, or *bleatings*, is the fluid secreted by the cow during the time from which she becomes dry to calving, and shows very great difference to ordinary milk in its composition, being much richer in albumenoids and poorer in milk-sugar and fat. In colour it is generally reddish yellow, due chiefly to blood corpuscles; the colour soon passing to the yellow colour of ordinary milk. On heating, it coagulates to a thick jelly-like mass, due to the large amount of coagulable albumenoids present, viz., globulin and albumin. In its chemical composition it differs chiefly from ordinary milk in the amount of globulin it contains, varying in amount from 5 to 18 per cent in the first milking, while milk contains from 0.01 to 0.2 per cent. Previously the albumin and globulin were estimated together, but since more exact methods for their separation have been found, it is as well to estimate both. The amount of albumin is small in comparison with the misleading figures of albumin + globulin previously given, as albumin varying from 0.0 to 2.0 per cent and the casein is not proportionately in such large quantities, varying from 2.75 to 5.35 per cent. The fat varies from 2 to 4.5 per cent, but soon regains its normal amount; the milk sugar is usually present in about the same quantities as the fat, and increases at about the same rate.

Since few complete analyses have been recently carried out with colostrum, the following investigation will no doubt add to the present knowledge of the composition of colostrum. The analyses were carried out in the laboratories of the Cheshire Agricultural College, Holmes Chapel, the colostrum being taken from a shorthorn cow—which calved on April 10, 1902, at 4 p.m.—twice a day, at 6 a.m. and 4.30 p.m. The following were determined in each sample:—

Specific gravity, dry matter, ash, fat, milk-sugar, total nitrogen, casein, globulin, and the albumin.

The separation of the various albumenoids is the most difficult and usually the most unsatisfactory from a purely chemical standpoint, since the only method available is fractional precipitation, and whether one is always able to completely get rid of the one before precipitating the other is an open question, and possibly the reason of such varied results by different investigators. Still, as most mixtures of albumenoids have been isolated by these means, and shown by ultimate analysis to be very different from each other, one may as well keep to the usual and (at present) only means of separation. An exhaustive investigation into the different methods of separating and determining

the albumenoids of milk has just been published by Gustav Simon (Dissertation, Halle), and from his research the methods giving the most correct results were adopted in the present investigation.

The specific gravity was determined by the pycnometer. For the total solids, 10 grms. of the sample were weighed into a platinum dish, and, after the addition of a few drops of alcohol to cause coagulation, evaporated on the water-bath to dryness, and then dried to constant weight in the hot-air oven at 105° C. The dried mass was carefully heated over a small flame till it had acquired a bright grey colour, and the residue weighed as ash.

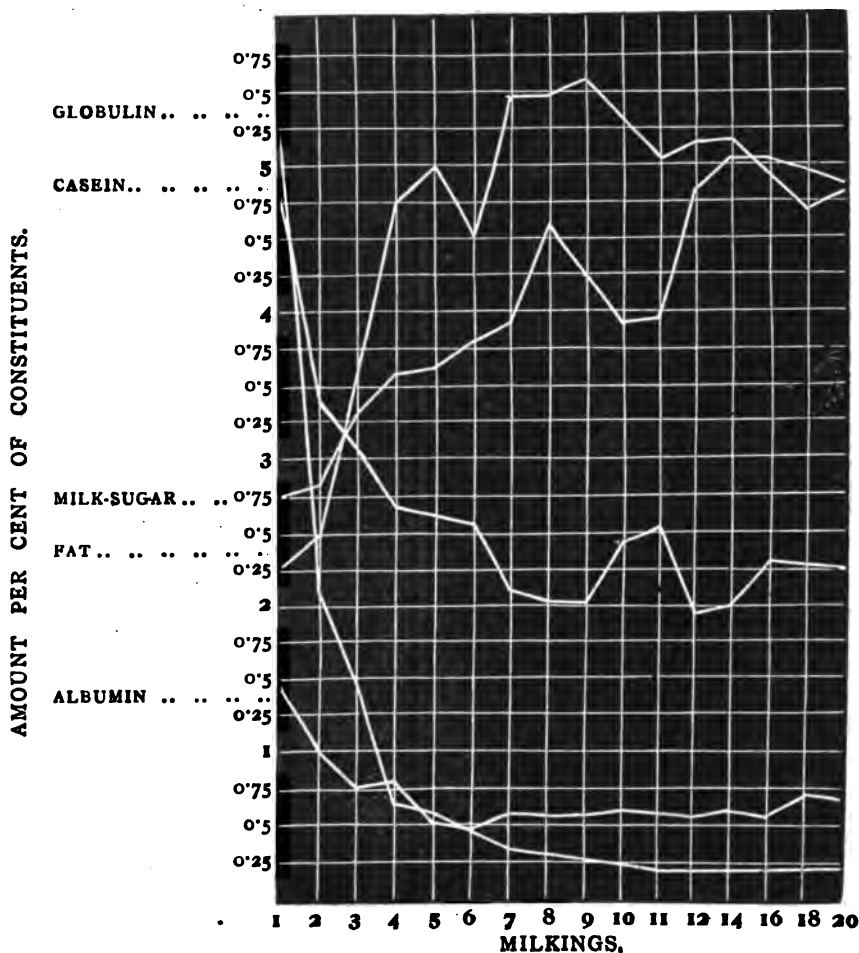
The fat was estimated by Adam's method, using 10 grms. of milk in each case.

For the milk-sugar, 25 grms. were diluted to about 400 c.c. in a 500 c.c. flask, and the albumenoids precipitated by Ritthausen's method, by adding 10 c.c. of Fehling's copper sulphate solution and enough 10 per cent sodium hydrate solution so that the solution still remained slightly acid (usually 7 c.c.). After settling, 100 c.c. of the clear liquid were then run into 50 c.c. of boiling Fehling's solution and boiled for about five minutes. The reduced cuprous oxide was then filtered through an Allihn's filter-tube, dried and reduced in a current of hydrogen, and weighed. From this the milk-sugar was calculated.

The three albumenoids were determined by diluting down 10 grms. of the colostrum to 100 c.c. with water; this being necessary, especially in the first few samples, in order to bring into solution the fairly large amount of suspended albumin. For the precipitation of the casein there are several methods recommended, but the one giving the best results is that of Schlossmann, who uses a saturated solution of alum, since an excess of this reagent does not re-dissolve to any extent the casein. To the diluted colostrum, warmed to 40° C., were added, drop by drop under continued shaking, the alum solution until a flocculent precipitate came down. After cooling, the precipitate was filtered off and washed with distilled water, and the nitrogen estimated by Kjeldahl's process, and the result multiplied by the factor 6.37 taken as casein.

The filtrate was saturated with moistened magnesium sulphate crystals, the precipitated globulin filtered off, and, after washing with saturated magnesium sulphate solution, the nitrogen was estimated. The albumin in the filtrate was precipitated by Almén's tannic acid solution, viz., 4 grms. tannic acid, 8 c.c. 25 per cent acetic acid, and 190 c.c. 45 per cent alcohol. Of this solution, 15 c.c. were used each time, and the nitrogen estimated in the precipitate in the usual way. (In each case the amount of nitrogen contained in the filter-paper used was deducted).

No. of milking.	Dry matter.	Ash.	Fat.	Lactose.	Total albumin.	Casein.	Globulin.	Albumin.	Spec. grav.
1.	22.878	1.034	2.302	2.742	12.236	4.858	5.3206	1.454	1.068
2.	16.232	0.874	2.490	2.552	6.976	3.353	2.048	1.014	1.037
3.	15.158	0.866	3.410	3.376	5.829	3.099	1.451	0.758	1.035
4.	15.902	0.824	4.748	3.628	4.652	2.705	0.662	0.782	1.030
5.	15.738	0.820	5.102	3.636	4.018	2.618	0.558	0.524	1.030
6.	15.748	0.818	4.554	3.862	4.042	2.5631	0.488	0.499	1.029
7.	15.718	0.804	5.496	3.922	3.464	2.211	0.316	0.627	1.029
8.	15.624	0.802	5.470	4.576	3.360	2.176	0.272	0.610	1.030
9.	15.476	0.824	5.626	4.220	3.354	2.150	0.255	0.591	1.030
10.	16.016	0.860	5.372	3.782	3.480	2.427	0.246	0.620	1.031
11.	15.970	0.846	5.048	3.820	3.524	2.520	0.2293	0.597	1.031
12.	16.102	0.842	5.152	4.800	3.062	1.965	0.212	0.554	1.031
14.	16.550	0.844	5.156	5.008	3.216	2.201	0.203	0.562	1.031
16.	16.282	0.836	4.902	5.010	3.328	2.341	0.194	0.554	1.031
18.	16.094	0.820	4.708	4.902	3.314	2.322	0.194	0.571	1.031
20.	16.058	0.814	4.796	4.874	3.242	2.250	0.198	0.562	1.030



For the total nitrogen 5 grms. of the colostrum were taken. The results are given in the accompanying table.

The gradual rise and fall of some of the constituents is strikingly shown by the diagram. In this diagram it is seen that the globulin falls very rapidly for the first few milkings, then soon reaches a constant amount. The quantity of globulin in the later sample does not agree with the figures given by Fleischmann for milk ("Lehrbuch der Milchwirtschaft," p. 30), but as in every case a

fairly large precipitate was obtained with magnesium sulphate, I must stand by the results obtained. It will be seen that the fat and milk-sugar increase, while all the albumenoids decrease; the rise and fall respectively of the milk-sugar and casein corresponding with remarkable exactness.

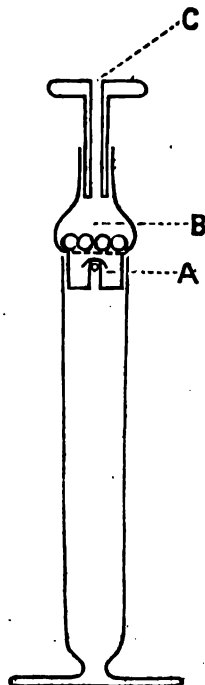
Equilibrium appears to be reached after the ninth milking, and for ordinary purposes the milk could be used after this period.

AN APPARATUS FOR THE DETECTION AND DETERMINATION OF MINUTE TRACES OF ARSENIC.

By EDWIN DOWZARD, F.C.S.

THE accompanying sketch (half actual size) is that of an apparatus for the detection of minute quantities of arsenic.

Using this apparatus 1/10,000,000 grm. can be detected, and 1/15,000,000 to 1/20,000,000 grm. can just be recognised. For all practical purposes 1/4,000,000 grm. may be taken as the limit. The apparatus is used as follows:—A rod of pure zinc, 2 c.m. long and 5 m.m. in diameter, is placed for about half to one minute in boiling water containing about 5 per cent pure HCl (sp. gr. 1.124); this is to remove any surface impurities. The rod is then washed in distilled water, and placed in the apparatus.



A, Trap.
B, Scrubbing chamber containing beads.
C, Orifice, 3 m.m. in diameter.

Two c.c. of HCl (sp. gr. 1.124), free from arsenic, and containing 6 drops of a 15 per cent solution of cuprous chloride in 100 c.c., is mixed with the substance to be tested, which must be in solution, and the liquid made up to 8 c.c. Before introducing the liquid a piece of Swedish filter-paper, 3 c.m. square, is prepared as follows:—A ring about 12 m.m. in diameter is drawn with a graphite pencil in the centre of paper, which is then moistened inside the ring with 5 per cent mercuric chloride solution, and dried. The ring is drawn merely to show the position of the mercuric spot. The glass beads in the scrubbing chamber may be moistened with a solution of lead acetate or cuprous chloride; the trap underneath is to prevent any washing liquid from entering apparatus, or the scrubbing chamber may be filled with cotton-wool which has been saturated with a 5 per cent solution of lead acetate, and dried.

The mercuric chloride paper is now attached to the top of apparatus with a clip, so that the orifice is under the centre of ring. The liquid to be tested is introduced into apparatus, which is placed in water at 60° C. (the water

should be on the same level as the liquid inside apparatus). After thirty minutes the paper is removed and examined in full daylight; a minute trace is indicated by a lemon-yellow spot and a trace by a deep orange-brown spot.

A blank test with the reagents must precede the examination. It is only necessary to test the same batch of acid and zinc occasionally.

In the absence of organic matter this test is of extraordinary delicacy; for instance, if the sample is a liquid and 4 c.c. is used, 1 part in 16,000,000 can be detected. If the sample is a solid and 0.5 grm. is used, 1 part in 2,000,000 can be detected.

Organic substances and liquids containing organic substances should be treated as follows:—

Solids.—0.5 grm. is made into a paste or strong solution with water, and mixed with 0.2 grm. of pure calcium oxide free from arsenic; the mixture is then dried on an asbestos felt and ignited; this should be done in a small porcelain dish. The residue is mixed with a few c.c. of water, and HCl added drop by drop until solution is effected; this solution is then examined as described above.

Liquids.—Two c.c. is mixed with 0.1 grm. of pure calcium oxide free from arsenic, and the mixture treated as in the case of solids.

If the sample to be examined contains sulphites or hypophosphites it should be treated as follows:—The substance should be dissolved in water or dilute HCl, a slight excess of bromine water added, and the mixture heated until free from bromine; the solution is then cooled, and treated as before described. This apparatus is recommended for the examination of beer and glucose for arsenic; the lime treatment should be followed. To facilitate comparisons with the standards, the yellow spots should be encircled with a black band about 2 m.m. wide, made with a graphite pencil; the inner edge of the band should touch the outer edge of the spot.

In the quantitative determination of minute traces of arsenic by this method the following points should be observed:—

1. The amount of HCl used, the final bulk of the liquid, and the size of the zinc rod should always be the same. The starting temperature should be 60° C.
2. The reaction should always occupy thirty minutes.
3. When comparing the sample with known amounts of arsenious acid; the standard test must contain the same weight or volume of the same substance as sample, but free from arsenic.

A standard set of spots may be prepared for each substance; they should be kept in a tightly stoppered bottle in a dark dry place.

The cuprous chloride solution is prepared as follows:—Sixteen grms. of pure CuO are dissolved in 110 c.c. of HCl, 13 grms. of thin copper-foil are cut into small pieces, and boiled with the above solution for twenty-five minutes; the resulting solution of cuprous chloride is poured into 1000 c.c. of distilled water, and the white precipitate washed by decantation. The chloride is then dissolved in HCl free from arsenic, and the solution standardised to contain about 15 grms. of cuprous chloride in 100 c.c.

The apparatus described above is made by Messrs. Gallenkamp and Co. 19, Sun Street, London.

A Method for the Volumetric Estimation of Silver.

—L. W. Andrews.—To the solution containing the silver to be estimated, slightly acidulated with nitric acid, a ferrous as well as a ferric salt is added; at least as much iron in each of these two salts as there is silver to estimate. Then, by means of a graduated burette, a titrated solution of iodine is added, coloured blue with starch; this solution should be added gradually with frequent stirring. The burette is read off at the moment when the colouration persists. The reaction is as follows:— $2\text{NO}_3\text{Ag} + 2(\text{NO}_3)_2\text{Fe} + \text{I}_2 = 2\text{AgI} + (\text{NO}_3)_3\text{Fe}_2$. — *Zeit. Anorg. Chem.*, vol. xxvi, p. 175.

DOUBLE SALTS IN SOLUTION.

By P. N. EVANS.

THE writer's attention was recently called to the fact that some electrolytes in saturated aqueous solution are not precipitated by the addition of a second electrolyte having an ion in common with the first. As on further investigation several instances of this kind were noted, an explanation was sought; some of the facts have been observed by others, but no attempt made to account for them. A reasonable solution of the difficulty is, it is thought, here presented.

The case may be briefly stated as follows:—When an electrolyte capable of dissociating into two ions is dissolved in water, the proportions existing between the ionised and non-ionised molecules is supposed to be represented, according to Van der Waal's equation, by the expression $a.b = k.c$, in which a , b , and c are the concentrations of the anions, kathions, and non-ionised molecules of the electrolyte respectively, and k a constant depending on the nature of the electrolyte, and independent of the concentration for moderately or highly dilute solutions.

Supposing this equilibrium to have become established, which is the case in an exceedingly brief time, if not instantaneously, any addition of either kind of ion concerned, the quantity of solvent remaining the same, must result in an increased value for its concentration, and produce a corresponding increase in the number and concentration of the non-ionised molecules; for, k being a constant, any increase in the value of a or b will involve an increase in that of c if the equilibrium is to be maintained.

If to a saturated solution of an electrolyte there be added a second soluble electrolyte having an anion or a kathion in common with the first, there must result a state of supersaturation with regard to the first electrolyte or a separation of a portion of it in solid form.

Many examples of this are familiar to all chemists. For instance, a saturated solution of sodium chloride is instantly precipitated by the addition of concentrated hydrochloric acid, in spite of the water that is added at the same time; this case is complicated and the precipitation assisted by the chemical union taking place between the hydrochloric acid and some of the water, made evident by the evolution of heat, thus increasing all the concentrations by removing (chemically changing) some of the solvent. The same result is obtained and the same reasoning applies when calcium chloride is added to a saturated solution of sodium chloride. The precipitation of sodium chloride is brought about without this complication of causes by the addition of crystallised potassium chloride and by anhydrous sodium sulphate, and even crystallised sodium sulphate, ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$), gives the same result in spite of the water added in the crystals.

Similarly, potassium chloride can be precipitated by hydrochloric acid (HCl), sodium chloride (NaCl), or potassium sulphate (K_2SO_4); and copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) by cupric chloride ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$), copper nitrate ($\text{Cu}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), or sulphuric acid (H_2SO_4); barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) by hydrochloric acid or sodium chloride; calcium sulphate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) by sulphuric acid, potassium sulphate, calcium nitrate ($\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$); lead chloride (PbCl_2) by hydrochloric acid, sodium chloride, potassium chloride.

In this list we have a very wide range of solubility:—100 parts of water dissolve NaCl , 35; KCl , 32; $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, 40; $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, 33; $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, 0.20; PbCl_2 , 0.74 parts. It is not evident whether easily or difficultly soluble substances should respond more readily in yielding a precipitate under these conditions, for the much greater degree of supersaturation attainable with an easily soluble substance, provided precipitation does not take place, may be offset by the greater difficulty in disturbing this state of supersaturation and causing the separation of a solid.

The greater the solubility of the electrolyte added to

the saturated solution, the more readily a precipitate should be obtained; and the higher the ionisation constant of the second electrolyte, the more probable is a marked result.

In spite of the apparently favourable conditions in this respect, all attempts to precipitate lead chloride from its saturated solution by means of lead nitrate were fruitless. A saturated solution of the chloride is very easily prepared, owing to the great difference in solubility in hot and in cold water, by warming the solution in contact with an excess of the salt, and then cooling and decanting. The immediate and copious precipitate produced in this solution by the addition of sodium or potassium chloride or hydrochloric acid seems to indicate that the tendency to remain in the supersaturated condition is very slight in the case of this salt, yet the addition of lead nitrate crystals ($\text{Pb}(\text{NO}_3)_2$) to the solution, even in the quantity made possible by the ready solubility of the nitrate (48 parts in 100), fails to cause any precipitation, either immediately or on long standing, or even on adding a crystal of lead chloride to induce crystallisation from the solution, supposing it to be supersaturated. Lead nitrate, like most normal salts, has a high ionisation constant, more than half that of the strongest acids in a 0.1 per cent solution—calculated by Arrhenius from conductivity experiments by Kohlrausch. This fact, and its ready solubility, should be most favourable to the precipitation of the lead chloride, on account of the considerable increase in the concentration of the lead ions made possible thereby.

In harmony with the usual similarity of barium to lead, a saturated solution of barium chloride shows no sign of precipitation with barium nitrate ($\text{Ba}(\text{NO}_3)_2$); as already stated, a precipitate is produced by the addition of hydrochloric acid, or sodium or potassium chloride, to the saturated barium chloride solution.

Similarly, potassium sulphate, though precipitated from its saturated solution by the addition of potassium chloride or sodium sulphate, gives no precipitate with sulphuric acid; and calcium sulphate is not thrown down by either sodium sulphate or ammonium sulphate, but does separate slowly on the addition of potassium sulphate, and more quickly with sulphuric acid.

Apparently, then, in these cases, the number of non-ionised molecules of the first is not increased by the addition of the second electrolyte, and the only plausible explanation seems to be that in these cases double salts are formed, namely, lead chloride-nitrate (PbClNO_3), barium chloride-nitrate (BaClNO_3), hydrogen potassium-sulphate (HKSO_4), calcium-sodium sulphate ($\text{CaNa}_2(\text{SO}_4)_2$), and calcium-ammonium sulphate ($\text{Ca}(\text{NH}_4)_2(\text{SO}_4)_2$). The simplest formulae are here suggested, but of course the proportions of the two acids or two bases in the molecule may not be as represented in these.

Taking the case of lead chloride as an example, the addition of the lead nitrate must immediately increase the number of lead ions, but at the same time diminishes both this and the number of the chlorine ions by permitting the formation of lead chloride-nitrate, so that the value of the product of the concentrations of lead and chlorine ions is not thereby increased, thus causing no rise in value of the concentration of the lead chloride, and therefore no separation of this substance as a precipitate. It does not follow, however, that this peculiarity of behaviour must accompany the formation of a double salt under similar circumstances, for it may be that the increase of the concentration of one kind of ion concerned may more than counterbalance the simultaneous decrease in this and that of the other kind of ion involved; this is simply a question of the ionisation constants of the electrolytes present.

It seems reasonable to suppose that double salts should exist whenever there is a polyvalent acid in presence of two or more bases, or a polyvalent base in presence of two or more acids, though other evidence of the existence of most of these compounds is at present lacking. Since, however, we commonly know and recognise the existence

of only those double salts which separate out from solutions of their constituents rather than these constituents themselves in distinct crystals, the evidence so far accepted for the existence of such compounds is of a very limited kind, namely, only their solubility relative to that of their constituents. In other words, if these separate in preference to the double compound, the latter does not exist so far as this kind of evidence is concerned.

Now supposing that in a solution of equivalent quantities of the constituents, the ionisation constants of these and the double salt are such that approximately equal numbers of molecules of the three kinds exist in the non-ionised condition, and the solution be concentrated, the double salt will separate if its molecular solubility (solubility divided by molecular weight) is less than that of either constituent, but not otherwise. Of course it is not probable that the constants are such as to more than approximately produce a condition like that imagined in the example just described; but inasmuch as normal salts do not differ very widely in their ionisation constants, the facts may be nearly enough in harmony with the supposition to make a study of these molecular solubilities not without interest in this connection, though the data available are not so numerous as desired.

An examination of the solubilities of twelve double salts selected at random shows the facts to be in accordance with this theory without a single exception, namely, that the molecular solubility of the double salt is less than that of either constituent. The figures are given in the following table, the formulæ being those of the substances separating out as crystals from their solutions:—

Formula.	a = molecular weight.	b = solu- bility in 100 parts water.	b ÷ a = molecular solubility (× 100).
K ₂ SO ₄	174	12.5	7.2
Al ₂ (SO ₄) ₃ .18H ₂ O ..	665	85	12.8
KAl(SO ₄) ₂ .12H ₂ O ..	474	9.5	2.0
(NH ₄) ₂ SO ₄	132	77	57.6
Al ₂ (SO ₄) ₃ .18H ₂ O ..	665	85	12.8
(NH ₄)Al(SO ₄) ₂ .12H ₂ O ..	452	9	2.0
K ₂ SO ₄	174	12.5	7.2
Cr ₂ (SO ₄) ₃ .18H ₂ O ..	717	120	16.7
KCr(SO ₄) ₂ .12H ₂ O ..	500	20	4.0
(NH ₄) ₂ SO ₄	132	77	57.6
Cr ₂ (SO ₄) ₃ .18H ₂ O ..	717	120	16.7
(NH ₄)Cr(SO ₄) ₂ .12H ₂ O ..	478	over 12	over 2.5
K ₂ SO ₄	174	12.5	7.2
Fe ₂ (SO ₄) ₃ .9H ₂ O ..	562	over 80	over 14.1
KFe(SO ₄) ₂ .12H ₂ O ..	503	20	4.0
(NH ₄) ₂ SO ₄	132	77	57.6
FeSO ₄ .7H ₂ O	278	60	21.6
(NH ₄) ₂ Fe(SO ₄) ₂ .6H ₂ O ..	392	17	4.3
NH ₄ Cl	53.4	33	61.8
SnCl ₄ .5H ₂ O	350	over 1900	over 543
(NH ₄) ₂ SnCl ₆	366	33	9.0
K ₂ SO ₄	174	12.5	7.2
CaSO ₄ .2H ₂ O	172	0.205	0.12
CaK ₂ (SO ₄) ₂ .H ₂ O ..	328	0.25	0.08
K ₂ SO ₄	174	12.5	7.2
CoSO ₄ .7H ₂ O	280	58	20.7
K ₂ Co(SO ₄) ₂ .6H ₂ O ..	437	19	4.3
K ₂ SO ₄	174	12.5	7.2
NiSO ₄ .7H ₂ O	281	67	23.9
K ₂ Ni(SO ₄) ₂ .6H ₂ O ..	437	5.3	1.2
KCl	75	32	42.6
MgCl ₂ .6H ₂ O	203	130	64.0
KMgCl ₃ .6H ₂ O	278	64.5	23.2
K ₂ CO ₃	138	110	80.0
Na ₂ CO ₃ .10H ₂ O	286	21	7.3
KNaCO ₃ .6H ₂ O	230	13	5.6

Purdue University, Lafayette, Indiana, U.S.A.,
April, 1900.

ON THE RELATIONS OF SULPHUR AND IODINE, AND THE IODIDES OF SULPHUR.

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OUR knowledge of the relations of sulphur and iodine, and of the so-called iodides of sulphur, is in a somewhat confused state. In many of our text-books we read that the blackish-grey indistinctly crystalline body formed when a mixture of the elements in equal atomic proportions is heated, even under water, is the mono-iodide, S₂I₂. Now, as a matter of fact, this product entirely lacks the chemical characteristics which analogy with the chloride and bromide indicates would be possessed by a true iodide. Gay-Lussac in 1813 (*Ann. de Chim. et de Phys.*, lxxviii., 308) states that any union between sulphur and iodine must be of the most feeble kind; indeed, that doubt might be placed on the possibility of two bodies so akin in nature and properties entering into true chemical combination at all. I hope to show that the bulk of evidence to date fully endorses the view thus expressed over ninety years ago.

The melting-point of the "blackish-grey material"—which, by the way, is 66°, and not 60° as given in the text-books—being so much below that of either of the elements composing it is the only strong fact in favour of its being a true chemical combination, but, remarkable to say, in all its other properties it more resembles a mixture formed by the solution, or may be partial solution, according to circumstances, of the one element in the other, and the slight evolution of heat which is said (but never yet proved) to take place when the act of union occurs might be attributable to this process of mere solution no less than to actual chemical combination.

It is now some twenty-eight years since I sent a communication on the subject of iodide of sulphur to the *CHEMICAL NEWS* (vol. xxx., p. 179), in which I gave the results of some experiments which had led me to doubt the compound nature of the product formed by simply melting a mixture of sulphur and iodine. I particularly noted that not only did the material readily yield up the whole of its iodine to such solvents as alcohol (preferably containing some ethyl iodide to increase the solvent power of the spirit) and a solution of potassium iodide, but that the sulphur left behind was in a crystalline form, and therefore readily soluble in carbon disulphide. To this latter fact I at the time attached some importance, it having been shown long before by Berthelot that sulphur separated from compounds, such as S₂Cl₂, wherein it acts as the positive radicle, is not soluble in the disulphide.

Later experiments, carried out at irregular periods and in widely different places, have more than confirmed the impression made on my mind so long ago, viz., that no definite iodide of sulphur is formed when a mixture of sulphur and iodine is simply heated to fusion. In all my efforts to form S₂I₂ in this way I exercised the utmost care in regulating the temperature at which the process of fusion was effected so as to keep it as low as possible, and thereby prevent the dissociation of any iodide that might be formed. The product obtained, however, when dissolved in CS₂ invariably gave a solution which on slow evaporation yielded successive crops of crystals containing variable percentages of sulphur. Thus in one series of operations I obtained the following results:—

First crop of crystals contained	0.38 per cent S
Second	1.09 ..
Third	3.51 ..
Fourth	38.56 ..

The final residue left after the evaporation of the solvent contained about 80 per cent of sulphur. When digested in KHO or NaHO the substance gave up the whole of its iodine, leaving all the sulphur behind in rhombic form, and no sulphite or thiosulphates were found in the solu-

tion, as would assuredly been the case had any S_2I_2 been present:—



The efforts made by chemists in different countries to form definite iodides of sulphur by bringing the constituent elements into close molecular association by mixing their carbon disulphide solutions have led to most contradictory results. Lamers (*Journ. Prakt. Chem.*, 1861, lxxxix., 349) obtained "what he believed to be sulphur hexaiodide, SI_6 , in the following manner:—Solutions of sulphur and iodine in various proportions in carbon disulphide were allowed to gradually evaporate at low temperatures, when crystals of the compound invariably separated out, no matter what the relative proportions of the two elements in the solution had been; unless, indeed, one or other of them, being in great excess, separated first before the hexaiodide crystallised out." G. vom Rath (*Pogg. Ann.*, cx., 17) submitted some crystals of the supposed new iodide to crystallographic examination, and was disposed to regard them as being isomorphous mixtures of sulphur and iodine, only he thought these elements were from his standpoint too dissimilar to form such mixtures. McLeod (*Brit. Assoc. Reports*, 1892, p. 690), and several other authorities, including Linebarger (*Amer. Chem. Journ.*, xvii., 33–59), have, however, clearly shown that the supposed SI_6 obtained by Lamers is merely a mixture of the two elements. In several trials made on the lines of Lamers' work I also failed to form the compound. Schlagdenhaufen (*L'Union Pharmaceutique*, civ., 1874) says:—"When solutions of sulphur and iodine in carbon disulphide are mixed, a coloured solution is obtained no matter what the relative quantities of the two elements may be; no heat is evolved, hence no action—no combination; it is a simple mixture of the metalloids." In this connection it is noteworthy that Ogier (*Comptes Rendus*, 1881, xcii., 9, 22) determined the heat of combination and of solution in carbon disulphide of a compound of sulphur and iodine, "which was stated to be S_2I_2 , and to have the character of a definite body"; he found the first to be quite too small to be taken into account, and the second to be equal to the sum of the heats of the two elements alone in the solvent. Linebarger (*Amer. Chem. Journ.*, xvii., 33–59) points out that Ogier does not indicate the degree of concentration of the solutions of sulphur and iodine which he mixed, and therefore the only legitimate conclusion to be drawn from his statements is that in dilute solutions no combination between the elements takes place. Working with concentrated solutions containing sulphur and iodine in equal atomic proportions he obtained by fractional crystallisations four crops of crystals, each of which on analysis proved to have the composition S_nI_n . The solutions invariably gave this result, provided the evaporation was carried out very slowly and at a temperature of 10–15°. Linebarger, however, through having no thermo-chemical apparatus at hand, was unable to determine the heat evolved (if any) by the union of the elements in his concentrated solutions so to compare its amount with the sum of the heats of sulphur and iodine separately in the CS_2 . Neither did he make any crystallographic examination of his S_nI_n , but formed the opinion that the crystals were "rhombic tablets more or less pronouncedly." This chemist concludes, after an apparently earnest and painstaking research, that there is only one definite compound of sulphur and iodine, and that its molecular weight is S_2I_2 ; its state of combination is very weak, and the elements seem to be in a condition of molecular rather than atomic union:—"There seems to be more tendency on the part of the sulphur and iodine atoms to form molecular aggregations than to combine with each other."

(My efforts to obtain crystals of this mono-iodide, or indeed any other definite iodide, from carbon disulphide solutions—whether concentrated or weak—were not successful. Adopting the system of fractional crystallisation, and taking all precautions with respect to the rate

of evaporation, I found invariably that each succeeding crop of crystals contained more sulphur than its predecessor, until finally a residue consisting chiefly of sulphur remained after the disappearance of the solvent. These results were, of course, in my opinion, due to the fact that the elements were not in actual chemical union in the solution, and that therefore the tendency of the less soluble iodine was to first separate out. A concentrated solution in which the elements were mixed in equal atomic proportions yielded crystals containing:—

First crop of crystals	=	0.42	per cent sulphur
Second	"	0.57	"
Third	"	3.07	"
Fourth	"	35.93	"

On allowing the mother-liquor to completely evaporate a residue containing over 80 per cent of sulphur remained. Treated with a solution of potassium hydrate, neither the crystals nor the residue yielded a sulphite or thiosulphate, but merely gave up their iodine, leaving behind rhombic sulphur).

Grosourd, and later Lamers (*Journ. Prakt. Chem.*, 1861, lxxxiv., 349) state that an iodide of sulphur having approximately the formula S_3I_2 is obtained as a reddish-brown amorphous mass by the action of sulphuretted hydrogen on an aqueous solution of $KCl.ICl_3$:—



I repeated the work of these chemists, and am inclined to think an iodide of sulphur is formed by the interaction of these substances, but if the passage of the gas is sufficiently prolonged, the final products of the reaction are hydrochloric and hydriodic acids and free sulphur. It is, however, interesting to note that much of this sulphur is insoluble in carbon disulphide, which, if we accept the Berthelot theory, would indicate that it had been in the first stage of the reaction in actual chemical union with iodine. The reddish-brown powder quickly loses its iodine on warming *in vacuo*, melts at 66°, and yields some thiosulphate with separation of sulphur when acted upon by a solution of KHO. I hope to deal further with this interesting reaction in a future paper.

F. Guthrie (*Chem. Soc. Journ.*, xiv., p. 57) by acting upon sulphur monochloride with ethyl iodide, preferably in hermetically closed tubes, and in the proportions shown in the equation—



obtained the moniodide "in the form of fine tabular crystals of the lustre of iodine and in a state of absolute purity." Although the synthesis of the compound would enable one to infer its exact composition, Guthrie, to further satisfy himself, submitted a quantity to analysis by heating in a combustion tube with KNO_3 and Na_2CO_3 , whereby the sulphur and iodine were converted respectively into KI and K_2SO_4 . The results obtained were:—

	Theory.	Found.
S	20.13	20.28
I	79.87	79.81
	100.00	100.00

It is much to be regretted that Guthrie did not carry his work further. The method of analysis adopted could throw no light on the true constitution of the crystals. It in no way helped to prove that S_2I_2 had actually been formed in accordance with the equation, and, indeed, later investigation has shown that the sulphur and iodine are liberated in elementary form and crystallised as mere mixtures. McLeod (*Brit. Assoc. Reports*, 1892, p. 690) found the product of the reaction to have more the nature of a non-metallic alloy than a definite iodide, while Snape (*CHEMICAL NEWS*, lxxiv., 27–29) found the crystals, which at first sight appeared homogeneous and dark, on microscopic examination, to consist of a mixture of opaque

iodine and transparent rhombic sulphur crystals, from which he extracted by means of ether the whole of the iodine. On slowly volatilising the iodine from a quantity of the powdered crystals Snape obtained a residue "equivalent to 20.16 per cent of sulphur, theory requiring 20.13," and points out that "whether chemical union has taken place or not, equivalent proportions of the sulphur and iodine necessarily constitute the residue of Guthrie's operations after the expulsion of the ethyl chloride." In preparing some of the crystals, I followed the instructions given by Guthrie in his paper, and found the product to have all the appearances therein stated, but on dissolving it in carbon disulphide and submitting the solution to slow evaporation I obtained successive crops of crystals which showed an irregularity in composition similar to that observed in the case of the product formed by the simple fusion of a mixture of sulphur and iodine. The powdered crystals completely surrendered their iodine to KHO, and in the solution I failed to detect either any sulphite or thiosulphates, while the sulphur left after the removal of the iodine was in the rhombic state.

The opinion expressed by Sestini (*Repertoire de Chimie Appliquée*, 1863, v., 481) may not be so far from the truth after all:—There exist a great number of compounds of sulphur and iodine obtained by fusion and solution, all being analogous in character to metallic alloys." In any case it is high time that the authors of some of our more modern text-books should make their references to the "iodides" of sulphur more in harmony with ascertained fact than they appear to have hitherto done.

In an early communication to the *CHEMICAL NEWS* I shall give the further results of my investigations, and hope to produce certain physico-chemical data which may make more easily understandable the true relations of sulphur and iodine.

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ESTIMATION OF THALLIUM IN THE THALLOUS STATE.

By V. THOMAS.

DURING the course of the research I have been carrying on for the last two years on the thalloses salts, I have had many occasions to estimate thallium in the thallose state. The method I constantly used consists in transforming the salts of the protoxide into salts of the sesquioxide. It has the advantage of great simplicity over the older methods which have been in use up to the present, besides being much more accurate.

The oxidising agent used is bromide of gold, or, to speak more accurately, bromo-auric acid. By reduction the bromide of gold loses three atoms of bromine, and gives a deposit of metallic gold which can be weighed, so that when the reduction is terminated two atoms of gold correspond to three atoms of thallium, according to the reaction $3\text{TlCl} + 2\text{AuBr}_3 = 3\text{TlClBr}_2 + 2\text{Au}$.

It follows that, taking 197 as the atomic weight of gold and 204 as that of thallium, we can immediately find the amount of thallose thallium existing in combination, by multiplying the weight of the reduced gold by—

$$\frac{3 \times 204}{2 \times 197} = 1.5533.$$

The salt of thallium I used to control this method was the protochloride. So as to obtain it in a satisfactory state of purity the commercial product was washed with cold water several times, then transformed into the trichloride, and finally reduced by means of sulphurous acid. The precipitated chloride was dissolved in boiling water, and the solution left to cool. The chloride thus deposited was then ready for analysis.

A known weight of the chloride, about 0.5 grm., was

dissolved in cold water. The solution was heated on the water-bath, and the solution of bromide of gold added. The reduction takes place immediately, or at the end of a few moments. The gold is precipitated in the form of a brown powder, which sometimes, but rarely, has a metallic lustre. After making certain that the amount of bromide of gold added is sufficient, which can be seen immediately by the colour of the solution, the whole is left in a warm place for eight or ten hours; the gold is then collected on a filter, dried, and weighed.

In this manner I found the proportion of thallium in the protochloride to be:—

I.	II.	III.	IV.	V.	Theory.
Tl = 85.02	85.29	85.16	85.30	85.18	85.21

These figures all agree within 0.25 per cent.

When the estimation has to be carried out on very small quantities the figures come out a little too high. In such a case, the amount of gold weighed being very small, it is probable that the gold reduced by the fumes in the laboratory during the time the solution is left to stand in a warm place, comes into account, although the amount is very small. I obtained the following results:—

TlCl taken.	Gold weighed.	Tl found.	Tl calculated.
0.2320	0.1280	85.69	85.21
0.2940	0.1620	85.58	—

Practically at least 0.2 or 0.3 grm. of gold should be weighed to obtain good results.

The bromo-auric acid used was prepared very easily by dissolving gold in bromised bromhydric acid. After solution it is evaporated on the water bath.

The bromo-auric acid is then deposited on cooling in long dark coloured needles, which are easily re-dissolved in water. The filtered solution is kept ready for use. When we use solutions which have been prepared for some time it is necessary to make certain that they are perfectly clear.

It often happens that after a long time solutions of bromide of gold deposit a yellowish-brown powder on account of a slight reduction which takes place. This powder, if we are not careful, may remain in suspension for a considerable time and remain unnoticed; it is therefore advisable to use solutions which have been recently filtered.—*Bull. Soc. Chim.*, Series 3, vol. xxvii, No. 11.

ON THE DOUBLE SALTS OF TETRATOMIC TITANIUM.

By A. ROSENHEIM and C. SCHÜTTE.

I. Chlorotitanic Acid.

RECENTLY distilled chloride, TiCl_4 , dissolves in well-cooled fuming hydrochloric acid, giving a deep yellow solution which undoubtedly contains TiCl_6H_2 , but this product has not yet been isolated. When treated with cold water the solution remains limpid for some time, and finally deposits gelatinous titanic acid. The same takes place with a solution of this latter substance in fuming nitric acid. $\text{Ti}(\text{OH})_4$ has also been added to a solution of hydrochloric acid gas in absolute alcohol or ether, both being kept thoroughly well cooled; the reaction is very brisk. These solutions were then kept boiling for two or three hours in a flask fitted with a vertical condenser; the very titaniferous filtered liquors were then evaporated to dryness under reduced pressure, and a very refrangible, deep yellow coloured oily liquid was obtained, undoubtedly containing TiCl_6H_2 , but it could not be isolated, as the product of the ethyliferous residue decomposed when an attempt was made to distil it *in vacuo*.

During its preparation, a yellowish-white precipitate was also formed by the action of $\text{Ti}(\text{OH})_4$ on the

hydrochloric acid in the ether; its composition was $\text{TiCl}_3\text{OH} + (\text{C}_2\text{H}_5)_2\text{O}$.

Chlorotitanate of Ammonium, $\text{TiCl}_6(\text{NH}_4)_2 + 2\text{H}_2\text{O}$.—Solid chloride of ammonium is added to the hydrochloric solution of TiCl_4 ; this is thoroughly stirred while being kept quite cold. At the end of twelve hours a slightly crystalline, bright yellow deposit is formed, which is washed with ether. This salt is very easily decomposed in moist air, and becomes white.

In the same manner we obtain the *chlorotitanate of pyridine*, $\text{TiCl}_6\text{H}_2 \cdot 2\text{C}_5\text{H}_5\text{N}$, in small yellow crystals, but this is prepared by using an alcoholic solution; the salts of *quinoline*, $\text{TiCl}_6\text{H}_2 \cdot 2\text{C}_9\text{H}_7\text{N}$, and of *aniline*, $\text{TiCl}_6\text{H}_4 \cdot 4\text{C}_6\text{H}_5\text{NH}_2$, are prepared in a similar manner.

Ammoniacal Tetrachloride of Titanium, $\text{TiCl}_4 \cdot 4\text{NH}_3$.—This is an amorphous powder of a deep-yellow to reddish-brown colour, and is precipitated when we pass a current of ammonia gas through a concentrated and well cooled solution of TiCl_4 in perfectly anhydrous ether. In the same way with *pyridine* in ethereal solution we get an analogous body, $\text{TiCl}_4 \cdot 6\text{C}_5\text{H}_5\text{N}$.

II. Bromotitanic Acid.

Bromide of titanium gives results similar to those furnished by TiCl_4 ; by reacting on an alcoholic solution of hydrobromic acid, the concentrated solution deposits a white crystalline powder consisting of $\text{TiBr}(\text{OH})_3 + 1\frac{1}{2}\text{H}_2\text{O}$. Under the same conditions as with chlorotitanate of ammonium we obtain a *bromotitanate of ammonium*, $\text{TiBr}_6(\text{NH}_4)_2 + 2\text{H}_2\text{O}$, in almost black, red crystals. With bromohydrate of pyridine in the presence of an excess of hydrobromic acid, in alcoholic solution, we obtain the *bromotitanate of pyridine* in crystals of the same colour, and having a metallic lustre; but if the acid is not in excess we obtain, on evaporation in the cold, yellow needles of $\text{TiOBr}_2 \cdot 3(\text{C}_5\text{H}_5\text{N} \cdot \text{HBr})$. Finally, ammonia-gas, passed through an ethereal solution of TiBr_4 , gives a brown amorphous body which is without doubt $\text{TiBr}_4 \cdot 6\text{NH}_3$.

III. Titanic Thiocyanate.

An ethereal solution of TiCl_4 , well shaken up with thiocyanate of lead and filtered, gives on evaporation, first of all yellow needles which are probably persulphocyanic acid, then very deep reddish-brown needles having a metallic lustre; these cannot be dissolved without decomposition, and the formation of persulphocyanic acid, &c.; it is doubtless a titanate polythiocyanate. $\text{Ti}(\text{OH})_4$ dissolves readily in an aqueous solution of CNSH, giving a red colouration.

IV. Sulphates of Titanium.

Titanic hydrate can be dissolved in a warm alcoholic solution of sulphuric acid, and the evaporated liquid gives a white amorphous deposit of *basic titanate sulphate*, $\text{SO}_4\text{TiO} + 6\text{H}_2\text{O}$.

A solution of $\text{Ti}(\text{OH})_4$ in concentrated sulphuric acid, being carefully treated with a concentrated aqueous solution of potassic sulphate, gives an abundant crop of needle-shaped crystals felted together; this is the double potassic sulphate, $3\text{SO}_4\text{TiO} \cdot 2\text{SO}_4\text{K}_2 + 10\text{H}_2\text{O}$. In a similar manner we obtain the corresponding ammoniacal salt, $\text{SO}_4\text{TiO} \cdot \text{SO}_4(\text{NH}_4)_2 + \text{H}_2\text{O}$.

V. Oxalates of Titanium.

By the evaporation of a solution of $\text{Ti}(\text{OH})_4$ with a solution of binoxalate of potassium or of ammonium we obtain needles which can be re-crystallised in water; they consist of $(\text{C}_2\text{O}_4\text{K}_2)_2\text{TiO} + 2\text{H}_2\text{O}$, and also large clinorhombic crystals of $[\text{C}_2\text{O}_4(\text{NH}_4)_2]_2\text{TiO} + \text{H}_2\text{O}$.

The solution of these salts gives, with just the right quantity of BaCl_2 , a crystalline precipitate of $(\text{C}_2\text{O}_4)_2\text{BaTiO} + 2\text{H}_2\text{O}$. The evaporation of a solution of $\text{Ti}(\text{OH})_4$ in an aqueous solution of $\text{C}_2\text{O}_4\text{H}_2$ does not give any crystals, but in alcoholic solution a white, micro-crystalline product is obtained, soluble in water and in alcohol; this is the *basic oxalate of titanium*, containing

alcohol of crystallisation, $\text{C}_2\text{O}_4\text{TiO} + \text{C}_2\text{H}_5\text{OH}$. In aqueous hydrochloric solution a white amorphous body is obtained, $(\text{C}_2\text{O}_4\text{TiO})_2\text{O} + 12\text{H}_2\text{O}$.

VI. Tartrate of Titanium.

From alkaline bitartrates, after evaporation of the solutions, we obtain a white powder of—



the salts of sodium and of ammonium contain $10\text{H}_2\text{O}$.

The simple tartrate is $(\text{C}_4\text{H}_6\text{O}_2)_2\text{Ti} + 4\text{H}_2\text{O}$; it is an amorphous mass, soluble in water, $[\alpha]_D = 140.8^\circ$. In the presence of alcohol we obtain $\text{C}_4\text{H}_4\text{O}_6(\text{TiO})_2\text{O} + 7\text{H}_2\text{O}$, insoluble in water, but soluble in acids and in ammonia.—*Zeit. Anorg. Chem.*, vol. xxvi., p. 239.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, Thursday, June 5th, 1902.

Prof. THORPE, C.B., F.R.S., Vice-President, in the Chair.

MESSRS. Steel, F. King, S. King, West, and F. M. Perkin were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Edgar Leeder Edlin, B.A., Marsh Parade, Newcastle, Staffs; George Felix Dudbridge Green, 9, Ross Road, South Norwood, S.E.; Arthur Francis Hosking, Roskear Villas, Camborne, Cornwall; Douglas K. Jardine, B.Sc., 20, Doune Terrace, Kelvinvale, Glasgow; Leonard Smith, 14, West Hill, Highbury, N.

The certificate of Mr. Herbert Purtan, London Chambers, Durban, Natal, authorised by the Council under By-law I. (3), was read.

Of the following papers, those marked * were read:—

*91. "The Action of Ungerminated Barley Diastase on Starch." Part I. By J. L. BAKER.

When the diastase from ungerminated barley is allowed to act at 50° on a solution of soluble starch, hydrolysis proceeds until at the end of an hour to an hour and a-half the constants of the dissolved matter are $[\alpha]_{D^{93}} = 160-165^\circ$ and $R_{93} = 60-65^\circ$. After this the reaction is a comparatively slow one, and if continued for ninety-six hours the reducing power is only increased to $R_{93} = 70$, the iodine reaction being still blue.

An examination of the products, after the reaction had proceeded for one and a-half to two hours, showed that a dextrin and maltose were the sole products. After twenty-four hours, evidence of the presence of glucose was obtained, the amount of this sugar apparently increasing in proportion to the time of conversion.

The dextrin, isolated by precipitation with alcohol from the products of the conversion, gave a blue iodine reaction, a specific rotation of $[\alpha]_{D^{93}} = 190-195^\circ$, and a reducing power of $R_{93} = 0.55-1^\circ$. The alcoholic solutions from which the dextrin was precipitated were collected and examined, and found to contain maltose, glucose if the conversion period was prolonged, but no trace of any dextrin of less complexity than described above.

Barley diastase acts on the above-mentioned dextrin very slowly, the constants of the solution after ninety hours at $45-50^\circ$ being $[\alpha]_{D^{93}} = 160^\circ$ and $R_{93} = 32^\circ$. The products, which give a blue iodine reaction, consist of maltose, unaltered dextrin, and glucose. The presence of glucose could be detected in eighteen hours, but after ninety hours the amount formed had only slightly increased. The action of malt diastase on this dextrin was more vigorous. After eighteen hours at 55° the solution, which no longer gave an iodine reaction, had the constants $[\alpha]_{D^{93}} = 149.3^\circ$ and $R_{93} = 50-51^\circ$. An examination of

the products showed that they consisted of maltose, a mixture of achroo-dextrins, and a considerable quantity of glucose.

Since barley diastase is without action on maltose, the glucose found in the products from the prolonged action of barley diastase on soluble starch must be derived from the dextrin. Barley diastase completely liquefies starch paste in two to three hours at 50°, the products consisting of the above described dextrin and maltose.

The dextrin obtained by the action of barley diastase on soluble starch or starch paste differs markedly from Nageli's "amylodextrin," which was re-investigated by Brown and Morris (*Trans.*, 1889, lv., 449). In consideration of its general behaviour and close relation to the parent substance starch, the author proposes to term the new dextrin *α*-amylodextrin.

DISCUSSION.

Mr. A. R. LING asked if the author had continued the reaction between barley diastase and soluble starch solution for a longer period than 144 hours, in fact until (when working with an excess of the diastase) the final point was reached. It had recently been shown by Mr. B. F. Davis and himself that by the action of the diastase from air-dried malt on starch paste below 60°, the whole of the starch was converted into maltose in forty hours. He had been struck by Mr. Baker's observation that maltose was practically the sole cupric-reducing compound formed in the early stages of the reaction between barley diastase and starch solution. With regard to the dextrin which the author had named *α*-amylodextrin, and which gave a blue iodine reaction, he could not help thinking, in view of what was known of starch-conversion products, that this dextrin (despite its apparent stability towards barley diastase) must have contained soluble starch. An indication of this was afforded by the behaviour of the dextrin towards malt diastase when the iodine reaction vanished.

Mr. BAKER, in reply, said that with regard to the suggestion of Mr. Ling and Dr. Thorne, that *α*-amylodextrin was a mixture of another dextrin and unaltered soluble starch, he was convinced that *α*-amylodextrin was an entity, from the fact that when this dextrin was acted on by barley diastase for eighteen hours at 50°, it split up into maltose and unaltered *α*-amylodextrin. If this residual *α*-amylodextrin was then subjected to the action of barley diastase under similar conditions, the same products were formed. This treatment had been repeated many times, and it did not seem possible that under such circumstances, soluble starch could escape the action of barley diastase.

*92. "The Decomposition of Chlorates. Part V. Potassium Chlorate in presence of Oxides of Manganese, and the Theory of Perchlorate Formation." By W. H. SODEAU, B.Sc.

In continuation of the previous work of the author, it was now shown that the amount of chlorine accompanying the oxygen evolved from potassium chlorate in presence of oxides of manganese was not increased on reducing the pressure under which the decomposition took place, whilst secondary reactions removing free chlorine from gaseous products have previously (*Trans.*, 1901, lxxix., 251) been experimentally shown to be greatly decreased on decomposition under reduced pressure. It was thus seen that no such secondary reaction occurred in the present case.

This excludes all theories which assume the cycle of changes to include the formation of a compound containing manganese and chlorine, or potassium and manganese, such as McLeod's well known theory. Oxygen being the only remaining constituent, it followed from this process of exclusion that the cycle of changes brought about by addition of oxides of manganese was confined to alternate oxidation and deoxidation (like the decomposition of hydrogen peroxide in presence of the same substances). It is probable that the formation of potassium perchlorate during the decomposition of the

pure chlorate by heat was due to an exothermic reaction independent of that which yielded chloride and oxygen. The non-production of perchlorate seemed to be the natural result of the addition of a substance (manganese dioxide) which brings about the transformation into chloride and oxygen at a temperature far below that at which the chlorate begins to give chloride and perchlorate.

DISCUSSION.

Prof. E. J. MILLS said that, in association with Mr. Stevenson, he had, some years ago, investigated the action of manganic oxide on potassic chlorate. They had not been able to find or prepare an oxide free from constitutional water, and any attempt to render the oxide anhydrous resulted in a loss of oxygen. Eventually, they used the oxide of Beilstein and Javorsky. Their work had reference chiefly to the question of catalysis, and they found that small quantities acted in proportion more energetically than large ones. It should be borne in mind that the composition of hot manganic oxide was affected by pressure. Altogether, it was a question whether the numerous difficulties besetting the investigation could be satisfactorily surmounted.

Mr. SODEAU, in reply, stated that in his examinations of the aqueous extracts of the residues, phenolphthalein was employed with precautions against carbon dioxide, and a very small fraction of the amount of alkalinity found by Prof. McLeod would therefore have been detected. The extracts were actually slightly acid, and the residual manganese peroxide rapidly absorbed potash from solution.

*93. "Studies in the Tetrahydronaphthalene Series. I. The Diazoamino-compounds of *ar*-Tetrahydro-*β*-naphthalene." By C. SMITH, B.Sc.

It has been shown by Bamberger and Bordt (*Ber.*, 1889, xxii., 625) that *ar*-tetrahydro-*α*-naphthylamine combines with diazonium salts, yielding azo-compounds.

The experiments of the author prove that *ar*-tetrahydro-*β*-naphthylamine behaves like a benzenoid amine, and yields diazoamino-compounds.

The following compounds have been prepared and examined:—

p-Toluenediazoaminotetrahydro-*β*-naphthalene, $\text{CH}_3\cdot\text{C}_6\text{H}_4\cdot\text{N}_2\cdot\text{NH}\cdot\text{C}_{10}\text{H}_{11}$, yellow crystals, m. p. 107°; readily soluble in alcohol and benzene, sparingly in petroleum. *o*-Nitrobenzenediazoaminotetrahydro-*β*-naphthalene, $\text{NO}_2\cdot\text{C}_6\text{H}_4\cdot\text{N}_2\cdot\text{NH}\cdot\text{C}_{10}\text{H}_{11}$, orange-yellow crystals, m. p. 134°. *p*-Nitrobenzenediazoaminotetrahydro-*β*-naphthalene, $\text{NO}_2\cdot\text{C}_6\text{H}_4\cdot\text{N}_2\cdot\text{NH}\cdot\text{C}_{10}\text{H}_{11}$, well-defined chocolate-brown crystals, melting at 179° with violent decomposition. *p*-Bromobenzenediazoaminotetrahydro-*β*-naphthalene, $\text{Br}\cdot\text{C}_6\text{H}_4\cdot\text{N}_2\cdot\text{NH}\cdot\text{C}_{10}\text{H}_{11}$, crystallises in lemon-yellow needles which decompose slightly on exposure to the air and melt at 134°. Diazoaminotetrahydro-*β*-naphthalene, $\text{C}_{10}\text{H}_{11}\cdot\text{N}_2\cdot\text{NH}\cdot\text{C}_{10}\text{H}_{11}$, well-defined, amber coloured crystals, m. p. 104°. *β*-Naphthalenediazoaminotetrahydro-*β*-naphthalene, $\text{C}_{10}\text{H}_7\cdot\text{N}_2\cdot\text{NH}\cdot\text{C}_{10}\text{H}_{11}$, crystallises in light yellow, lustrous plates, m. p. 137.5°. Tetrahydro-*β*-naphthaleneazo-*β*-naphthylamine, $\text{C}_{10}\text{H}_{11}\cdot\text{N}_2\cdot\text{C}_{10}\text{H}_6\cdot\text{NH}_2$, dark red lustrous crystals moderately soluble in hot alcohol, m. p. 130°. Tetrahydro-*β*-naphthaleneazo-*β*-naphthol, $\text{C}_{10}\text{H}_{11}\cdot\text{N}_2\cdot\text{C}_{10}\text{H}_6\cdot\text{OH}$, vermilion-red crystals with a green shimmer, m. p. 153°; it is insoluble in sodium hydroxide, and only sparingly soluble in hot alcohol.

DISCUSSION.

Dr. MORGAN pointed out that the mixed diazoamines of the tetrahydronaphthalene series are members of an extensive series of diazoamino-compounds having the general formula XN_2HY , which can be produced from one or other of the two pairs of generators, XN_2Cl , YNH_2 and YN_2Cl , XNH_2 , where X and Y are dissimilar aromatic nuclei derived from benzene, naphthalene (*Trans.*, 1902, lxxxi., 90), and tetrahydronaphthalene.

The genesis of these substances and their decomposition with mineral acids justify the belief that they are solid

solutions of the two compounds, $\text{XN}_2\cdot\text{NH}_2$ and $\text{YN}_2\cdot\text{NH}_2$.

94. "Experiments on Phosphorus Textroxide." By C. A. WEST, B.Sc.

The author gave an account of the preparation of phosphorus textroxide and of his attempts to determine its molecular weight. Methods based upon observation of the freezing- or boiling-points of a solution could not be used, no suitable solvent being found. At a red heat, under atmospheric pressure, the compound does not volatilise rapidly enough to allow of the determination of vapour density, but four concordant experiments at about 1400° led to the unexpected conclusion that the molecular formula is P_8O_{16} , that is twice what would have been expected from comparison with P_4O_6 and P_4O_{10} .

Some new determinations of the vapour density of phosphoric oxide at this temperature were also made. The mean of six concordant results gave 300.4 for the molecular weight P_4O_{10} requiring 284 .

95. "The Decomposition of Compounds of Selenium and Tellurium by Moulds and its Influence on the Biological Test for Arsenic." By O. ROSENHEIM, Ph.D.

A paper has recently been published (Maassen, *Arb. Kais. Ges. A.*, 1902, xviii., 475) on the biological test for arsenic and the formation of organic compounds of arsenic, selenium, and tellurium by moulds. The author has been occupied for some time on the same subject in its connection with the detection of arsenic in beer and sugar, and had intended to publish his results in a more complete form. In view of Maassen's publication it has, however, been thought advisable to give a short résumé of the results in this direction confirming those of Maassen.

The biological test for arsenic (Gosio, *Ber.*, 1897, xxx., 1024) is said to be characteristic of this substance (Abel and Bottenberg, *Z. Hyg.*, 1899, xxxii., 449). The test consists in the formation of gaseous organic arsenic compounds, possessing a characteristic garlic odour, produced by the vigorous growth of certain moulds (*Aspergillus*, *Mucor*, and *Penicillium*) in media containing arsenic, and permits of its detection where chemical methods fail. When applying this test to certain substances (beer and sugar) which contained selenium and arsenic, the author noticed a pronounced faecal odour different from that produced by arsenic alone. It was thought that this was due to the presence of selenium, and experiments with soluble selenium compounds confirmed this view. It was also found that tellurium compounds were decomposed by *Penicillium brevicaulis*, giving rise to the production of a very characteristic odour. Owing to the extreme sensitiveness of the test with regard to arsenic, it was thought necessary to employ absolutely pure compounds of selenium and tellurium. The odour produced by the decomposition of selenium compounds is of a very disagreeable character (somewhat like skatol or mercaptan), whilst the gases formed by tellurium compounds possess a pronounced garlic odour. The test is extremely sensitive; 0.01 m.grm. in 1 c.c. of liquid is easily demonstrated by a vigorous growth of the mould. Unlike arsenic, pure selenium and tellurium are not attacked by the mould. It was found best to perform the test by using the medium proposed by Abel and Bottenberg (*loc. cit.*), namely, sterilised bread-crumbs, to which the sterilised liquid in question is added after infection with the mould.

The author has drawn attention elsewhere (CHEMICAL NEWS, 1901, lxxviii., 277) to the influence of selenium on certain chemical tests for arsenic, and it is evident from the above results that in applying the biological test for arsenic, it is also necessary to take the possible presence of selenium (and tellurium) into consideration.

96. "Constituents of Gambier and Acacia Catechus." By A. G. PERKIN and E. YOSHITAKE.

The short paper on catechin, just published (Kostanecki and Tambor, *Ber.*, 1902, xxxv., 1867) renders it necessary to give the results of an investigation which has been in

progress for more than two years. The gambier catechu employed has been found to contain two catechins (b) and (c), and a third (a) has been isolated from the acacia catechu.

Catechin (b), $\text{C}_{15}\text{H}_{14}\text{O}_6\cdot 4\text{H}_2\text{O}$, air-dried, colourless needles, corresponds in its melting-point, $175-177^\circ$, with Gautier's (b) catechin (*Bull.*, 1879, xxx., 567), and gives, on fusion with alkali, phloroglucinol, protocatechuic acid, and an acid resembling acetic acid. The *disaxobenzene* compound (compare Etti, *Monats.*, 1881, ii., 552), $\text{C}_{15}\text{H}_{12}\text{O}_6(\text{C}_6\text{H}_5\text{N}_2)_2$, salmon-red needles, m. p. $193-195^\circ$; its *triacetyl* derivative, $\text{C}_{15}\text{H}_9\text{O}_6(\text{C}_6\text{H}_5\text{N}_2)_2(\text{C}_2\text{H}_3\text{O})_3$, orange-red needles, m. p. $253-255^\circ$; *pentabenzoyl* catechin, $\text{C}_{15}\text{H}_7\text{O}_6(\text{C}_6\text{H}_5\text{O})_5$, colourless needles, m. p. $151-153^\circ$, and *tetrabenzoyle* catechin, prismatic needles, m. p. $171-172^\circ$, have been studied. Molecular weight determinations by the cryoscopic method of the two latter compounds confirm these formulæ.

Catechin (c), $\text{C}_{15}\text{H}_{14}\text{O}_6$, air-dried, contains no water of crystallisation, and forms colourless prisms, m. p. $235-237^\circ$. It yields an *axobenzene* compound, $\text{C}_{15}\text{H}_{12}\text{O}_6(\text{C}_6\text{H}_5\text{N}_2)_2$, orange-red needles, m. p. $215-217^\circ$, the *acetyl* derivative of which melts at $250-253^\circ$, and on fusion with alkali gives phloroglucinol and protocatechuic acid. It has been found at present only in minute quantity.

Catechin (a), $\text{C}_{15}\text{H}_{14}\text{O}_6\cdot 3\text{H}_2\text{O}$ (or less probably $\text{C}_{14}\text{H}_{14}\text{O}_6\cdot 3\text{H}_2\text{O}$) air-dried, forms colourless needles, and corresponds in its melting-point, $204-205^\circ$ with Gautier's (a) catechin. Molecular weight determinations of the *pentabenzoyl* derivative (prismatic needles, m. p. $181-183^\circ$) coincide with this formula. The *axobenzene* compound, $\text{C}_{15}\text{H}_{12}\text{O}_6(\text{C}_6\text{H}_5\text{N}_2)_2$, needles, m. p. $198-200^\circ$, gives a *triacetyl* derivative, $\text{C}_{15}\text{H}_9\text{O}_6(\text{C}_6\text{H}_5\text{N}_2)_2(\text{C}_2\text{H}_3\text{O})_3$, orange-red leaflets, m. p. $227-229^\circ$. Fusion with alkali gave phloroglucinol, protocatechuic acid, and an acid resembling acetic acid.

Cyanomaclurin (*Trans.*, 1895, lxvii., 939), a constituent of *Artocarpus integrifolia*, has been found to contain a phloroglucinol group, and is probably isomeric with these catechins.

97. "The Decomposition of Oxalacetic Hydrazones in Aqueous and Acid Solution, and a new Method of Determining the Concentration of Hydrogen Ions in Solution." By H. O. JONES and O. W. RICHARDSON.

When oxalacetic hydrazone is heated with dilute acids it gives rise simultaneously to pyruvic hydrazone and carboxylic dioxide, and to pyrazolone carboxylic acid and water (Fenton and Jones, *Trans.*, 1901, lxxix., 92), the amounts of the former products being in the inverse order of the concentrations of the hydrogen ions in the solutions used. The hypothesis suggested to explain these observations was that the negative ion of the oxalacetic hydrazones underwent the first of the above decompositions when heated, whereas the undissociated molecule underwent the second.

Experiment shows that the results are better explained by supposing that the rate of decomposition into pyruvic hydrazone is proportional to the concentration of oxalacetic hydrazone, whereas the rate of formation of the pyrazolone carboxylic acid is jointly proportional to the concentration of the original substance and of the hydrogen ions.

The reaction was investigated in two ways, first the rate of evolution of carbon dioxide at a constant temperature (about 80°) was determined, and, secondly, the total amount of carbon dioxide given off at 100° was determined, in both cases water and sulphuric acid of different strengths being used. The results obtained agree with the requirements of the second hypothesis.

According to both theories, the amount of carbon dioxide produced satisfies the following equation,—

$$\log \frac{1}{1 - \frac{C_2}{[C_2]_\infty}} = \beta t,$$

where C_2 = concentration of pyruvic hydrazone (or amount

of carbon dioxide evolved) at any time t , $[C_2]_{\infty}$ its final concentration, and $\beta =$ a constant.

The hypothesis of the decomposition of the negative ion requires that β should decrease with increasing concentration of hydrogen ions, whereas the view here put forward requires that β should increase. Now it was found that β did increase with increasing concentration of hydrogen ions. This forms a crucial test between the two views.

A quick and easy method for determining the concentration of hydrogen ions in solution has also been worked out and tested.

Experiments have been made with the β -bromophenyl hydrazones of oxalacetic acid which decomposes in a similar way, and the results show satisfactory agreement with requirements of the theory stated above.

98. "The Dissociation Constants of Oxalacetic Acid and its Hydrazones." By H. O. JONES and O. W. RICHARDSON.

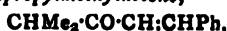
The authors have determined the dissociation constants of oxalacetic acid and its phenyl hydrazones. The conductivities of solutions of these substances were determined at 25°, and from the results the following values were obtained:—

For oxalacetic acid, $K = 1.33$. Oxalacetic hydrazone, $K = 0.11$.

99. "Derivatives of Butyrylpyruvic Acid." By A. LAPWORTH and A. C. O. HANN.

Certain points of interest arising out of the study of the Claisen reaction as applied to $\alpha\beta$ -unsaturated ketones (*Trans.*, 1901, lxxix., 1283) led the authors to study the action of ethyl oxalate and sodium on benzylideneisopropylmethylketone. The results were not those anticipated, but the following compounds have been prepared in the course of the work:—

Benzylideneisopropylmethylketone,—



is an oil (b.p. 274–276°). The oxime melts at 131–132° and the semicarbazones at 166–167°.

Ethyl isobutyrylpyruvate, $CHMe_2 \cdot CO \cdot CH_2 \cdot CO \cdot CO_2Et$, from isopropylmethylketone, ethyl oxalate, and sodium ethoxide, is a nearly colourless oil which boils and decomposes at 230–232°; it does not solidify at –15°. The copper, nickel, calcium, and other derivatives have been prepared. When the ester is hydrolysed with strong potassium hydroxide solution under certain conditions, and the resulting solution treated with excess of strong hydrochloric acid, a sparingly soluble compound is precipitated in quadratic plates. This is an acid potassium isobutyrylpyruvate having the composition $(C_7H_{10}O_4)_2 \cdot C_7H_5O_4K$; its solution gives a red coloration with ferric chloride, and becomes yellow on addition of excess of alkalis; it is only decomposed by a large excess of mineral acid; a similar sodium salt was not obtained.

Ethyl n-butyrylpyruvate, $Pr \cdot CO \cdot CH_2 \cdot CO \cdot CO_2Et$, is similar in properties to the ester described above. When it is hydrolysed with potassium hydroxide, it yields a sparingly soluble acid potassium n-butyrylpyruvate crystallising in asbestos-like needles, which has, however, the composition $(C_7H_{10}O_4)_2 \cdot C_7H_5O_4K$. As in the former instance, this substance is very stable towards acids, and no corresponding sodium salt could be obtained.

100. "Sulphocampholenecarboxylic Acid." By A. W. HARVEY and A. LAPWORTH.

When the residues obtained by evaporating the last aqueous mother-liquors in the preparation of ammonium bromocamphorsulphonate were fractionally extracted with alcohol, a small quantity of a new substance dissolved and was obtained in the form of transparent, calcite-like crystals. A small quantity of this product has also been obtained by Dr. Kipping.

The compound has the formula $C_{10}H_{15}SO_3NH_4$, and it is the hydrogen ammonium salt of an unsaturated, monocyclic, sulphocarboxylic acid, $CO_2H \cdot C_9H_{14}SO_3H$, for which the name *sulphocampholenecarboxylic acid* is proposed. That it is unsaturated is shown by its aqueous

solution instantly decolorising bromine water and an ice-cold solution of potassium permanganate.

The normal barium and calcium salts, $C_{10}H_{15}SO_3Ba$ and $C_{10}H_{15}SO_3Ca$, crystallise well and are more soluble in cold than in hot water; the normal ammonium salt is very soluble in water. The hydrogen barium and hydrogen calcium salts crystallise in fine needles. The chloride and bromide do not crystallise readily.

When solutions of the salts are treated with bromine, a molecular proportion of the latter is at once absorbed, and a very sparingly soluble substance separates. This appears to be a sultonecarboxylic acid,—

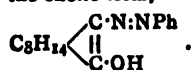


it crystallises from chloroform in small prisms, melts and decomposes at 155°, dissolves in alkalis and alkali carbonates, and is re-precipitated unchanged by acids.

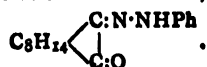
The investigation of the acid and its derivatives is in progress.

101. "Some Properties of Camphorquinonephenylhydrazones." By A. LAPWORTH and A. C. O. HANN.

It has been found by Betti (*Ber.*, 1899, xxxii., 1995) that camphorquinonephenylhydrazones ("camphormethylenehydrazones") usually exists as a mixture of two desmotropic forms. One of these melts at 180°, gives a reddish coloration with ferric chloride in benzene solution, and is therefore probably the enolic form,—



The other, melting at 155°, may be isolated by adding a trace of piperidine to a solution of the hydrazone in benzene; as it gave no distinct colour with ferric chloride, it was supposed to be the ketonic form,—



With Betti's permission, the authors have carefully studied the properties of the compound, and, although they are able to confirm all his statements with regard to the enolic form, they have not been able to isolate a second modification in the pure state, although there can be no doubt that one is present; they think that the substance melting at 155° must be a mixture of both forms.

The speed with which equilibrium between the two forms, in various media, is attained was investigated by observations of the mutarotation. Equilibrium appears to be reached most rapidly in alcoholic solution, a constant rotation being observed at once. In chloroform or benzene, however, the change occurs very slowly, as in 1 per cent solution at the ordinary temperature it occupies several days.

In benzene, with solutions varying in concentration from 0.1 to 1 per cent, the initial rotation, when the pure enol is used, is about $[\alpha]_D = +325^\circ$, and falls to between $+205^\circ$ and 290° according to the concentration. With all specimens of mixed material, even when prepared by Betti's method, a fall was invariably observed in dilute solution, indicating that the enol was still present in considerable amount.

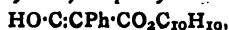
The effect of catalytic agents on the rate of change in benzene was carefully studied. Traces of bases, if not too weak, whether primary, secondary, or tertiary, accelerate the change, and, amongst others, ammonia, aniline, piperidine, pyridine, brucine, and strychnine were tried and found to be effective. Acids, on the other hand, retard the change, and it is possible to arrest the fall in rotation at any point desired by the addition of a minute quantity of trichloroacetic acid, when it will again proceed on addition of a trace of a base.

In all cases, as was to be expected, the state of equilibrium was found to be independent of the catalytic agent

102. "Optically Active Esters of β -Ketonic and β -Aldehydic Acids. Part I. *Menthyl Hydroxymethylphenylacetate*." By A. LAPWORTH and A. C. O. HANN.

The authors propose to investigate certain aspects of the question of tautomerism involving the migration of hydrogen atoms by the employment of esters of β -ketonic and β -aldehydic acids with optically active alcohols of high molecular weight. It is hoped that the compounds may thus be obtained in a crystalline, and therefore more easily purified form, and that by observation of the change of rotation, further evidence with regard to certain obscure points may be obtained.

Menthyl hydroxymethylphenylacetate,—



may be prepared by the action of sodium on a mixture of menthyl phenylacetate and formate dissolved in anhydrous ether. It is precipitated from the aqueous solution of its sodium derivative on addition of acids as an oil which rapidly solidifies, and crystallises from light petroleum in transparent prisms or pyramids, which belong to the tetragonal system. These melt at $82-83^\circ$, and exhibit triboluminescence, which is sufficiently brilliant to be observed in broad daylight. Its freshly prepared 2 per cent solution in chloroform has $[\alpha]_D = -74.6^\circ$; this falls in a few days to about -71° . In absolute alcohol, the rotation is $[\alpha]_D = -64.9^\circ$, and falls only slightly, if at all, in course of time. Although its alcoholic solution is rapidly coloured violet by ferric chloride, its solution in ether or benzene is not affected unless it is boiled with the salt for some time, or a trace of a base is added, when a similar coloration develops. The solid probably represents the aldehydic form of the ester (Wislicenus, *Ann.*, 1900, cccxii., 34).

The copper derivative crystallises in green prisms which melt and decompose at $92-95^\circ$; the sodium derivative forms thin, transparent plates.

The *phenylcarbamate*, $\text{NHPh}\cdot\text{CO}_2\cdot\text{CH}\cdot\text{CPh}\cdot\text{CO}_2\cdot\text{C}_{10}\text{H}_{19}$, crystallises in long, slender needles melting and decomposing at $235-237^\circ$. The *acetate*,—



separates from ethyl acetate in needles melting at $51-52^\circ$.

The oxime is immediately precipitated as an oil on mixing solutions of the ester with one of free hydroxylamine or hydroxylamine acetate. With phenylhydrazine, the ester is converted into the diphenylpyrazolone which is obtained from the corresponding ethyl ester.

The enolic modification of the ester appears to be very unstable, and under conditions in which the liquid enolic ethyl ester may be obtained, the above compound was always produced. No evidence of the existence of isomeric copper or acetyl derivatives could be obtained.

(To be continued.)

NOTICES OF BOOKS.

The New Volumes of the Encyclopædia Britannica. The Second of the New Volumes, being Vol. XXVI. of the Complete Work. London: *The Times*. Edinburgh: Adam and Charles Black. 1902. Pp. 763.

THIS, the second of the new volumes, includes all subjects whose titles come between A U S (Austria-Hungary) and C H I (Chicacole, a town of British India). In general appearance and contents this volume is quite up to the standard of excellence set by the first volume.

It includes, of course, many subjects which do not come within the range of the *CHEMICAL NEWS*; but, among the chemical and physical articles which appear, we must call special attention to the long and excellent essay on Bacteriology by Prof. H. Marshall Ward, D.Sc., F.R.S., who treats the subject in a general way; and the accompanying one by Dr. Robert Muir, who deals with the

pathological side of the question. Both these authors are known experts on the subject, and their names alone are sufficient guarantees of the value of the article. There are also two articles by Prof. H. L. Callendar, F.R.S., on Calibration and Calorimetry, both of which will well repay perusal; one on Cement, by Bertram Blount; and, most important of all, an article entitled "Chemistry," by Prof. H. E. Armstrong, F.R.S. This article fills about 40 pages of the *Encyclopædia*, and, long as it is, it is manifestly impossible to give more than a brief outline of this subject—the foundation of all the sciences—within such limits. However, Prof. Armstrong has treated the matter in a masterly manner, and has touched on practically every branch of the subject.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 23, June 9, 1902.

A New Isomerism of Asymmetric Nitrogen.—E. Wedekind.—The presence of two atoms of asymmetric nitrogen in a molecule appears to render possible the existence of the stereo-isomeric salts which take part in various reactions. The author endeavours actually to find new examples of such isomerism, and he proposes to apply Pope's method to the stable form which can be split up into two active isomers.

Benzene-azobenzoic Aldehyde.—P. Freundler.—The author showed in a preceding research that it is not possible to apply Friedel and Crafts' reaction to azobenzene or to its derivatives, or to prepare by this method compounds of the type $\text{C}_6\text{H}_5\cdot\text{N}=\text{N}\cdot\text{C}_6\text{H}_4\cdot\text{CO}\cdot\text{R}$. On the contrary, however, the synthesis of such compounds can be easily effected by applying to a mixture of nitrated derivatives the general method of preparation of the hydrazoic and azoic compounds:—



By this means benzene-azo- β -benzoic aldehyde can be obtained, $\text{C}_6\text{H}_5\cdot\text{N}=\text{N}\cdot\text{C}_6\text{H}_4\cdot\text{CHO}$. The author describes the formation of this compound and its principal properties. He now proposes to complete the investigation of the aldehydes, and to utilise the same reaction to prepare azoic derivatives with alcoholic and acid ketonic function.

MEETINGS FOR THE WEEK.

THURSDAY, 10th.—Institution of Mining and Metallurgy, 5. "The Diehl Process," by Hans C. Knutsen. "The Pierrefitte Concentrating Mill," by Mervyn S. Stutchbury.

ST. PAUL'S SCHOOL, West Kensington.—

An EXAMINATION will be held at the above School on Tuesday, September 16th, 1902, and the following days, for filling up Twenty or more Vacancies on the Foundation.—Full particulars can be obtained on application to the Bursar.

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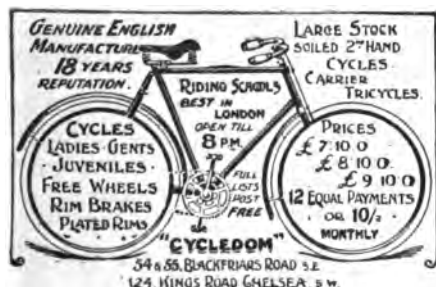
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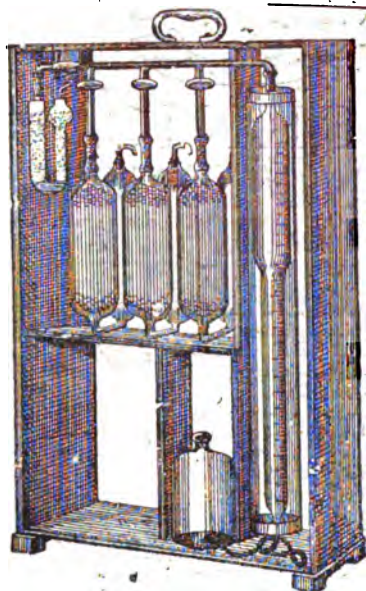
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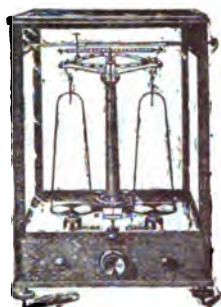
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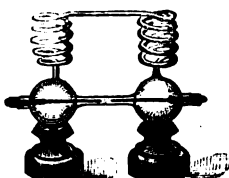
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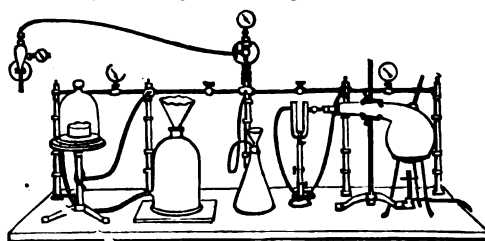
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THE CHEMICAL NEWS.

Vol. LXXXVI., No. 2224.

THE MELTING-POINT OF CHROMIUM.

By ERNEST A. LEWIS.

As the melting-point of chromium has not been determined accurately I obtained some chromium of 99 per cent purity, and free from carbon made by the Goldschmidt process by reduction of chromium oxide with powdered aluminium, and determined the melting-point with a Le Chatelier pyrometer.

I took a piece of chromium as big as a pea and put it in a hole in a lump of quicklime, and then directed the flame of a blowpipe fed with coal-gas and oxygen on to it; when the chromium was melted the thermo-junction of platinum and platinum-iridium was placed in it, and the point at which the spot of light of the galvanometer was stationary a few seconds was noted. This is very difficult, as the junction is very often broken by being put into the molten chromium at a high temperature.

Two experiments gave the melting-point of chromium to be:—

No. 1	1510° Centigrade
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Mean	1515° "

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Boiling-point of water	100° C.
Melting-point of zinc	419° C.
Melting-point of aluminium	657° C.
Melting-point of copper (in air)	1065° C.

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SANDALINA OIL AS A PRODUCT OF THE DECOMPOSITION OF GLUCOSIDES.

By Dr. T. L. PHIPSON.

THE name of sandalina has been given to a kind of petroleum which issues with the water from a spring at Santa Clara, in the island of Cuba. On account of the peculiar odour of the water it has been called the Sandalwood Spring. I have written to my friend Mr. Charles Thornton, C.E., to send me a sample of this water the first time he visits that neighbourhood; in the meantime, however, it has been examined by Mr. H. N. Stokes, who finds that the oil obtained is quite transparent and of the colour of amber. It has a specific gravity of 0.901, and a peculiar odour resembling that of cedar wood, and distills between 230° and 330° C. It appears, according to this chemist, to consist chiefly of naphthalenes. Naphthalene or petroleum acids, he says, are formed in notable quantity even in a rapidly conducted oxidation with chromic acid. The final products of oxidation are carbonic acid, acetic acid, and water (with traces of acetone and higher homologues).

Many years ago in examining certain glucosides discovered by me in the fruit of the walnut and the root of the strawberry (*Comptes Rendus* and *CHEMICAL NEWS*, 1869, and 1878), I observed the presence of a small quantity of an oily substance floating upon the liquid during the decomposition of these glucosides (regianine and fragarine) by boiling with dilute hydrochloric acid, and the odour of cedar wood was so strong that it diffused itself throughout the laboratory. Other glucosides, notably a remarkable one which I extracted some time ago from the *Ampelopsis*

(*Cissus quinquefolia*) gave a similar result, and I have little doubt that this odoriferous substance is the body now called sandalina; and just as Pasteur once told me that succinic acid is a constant product of the fermentation of sugar, it is probable that sandalina oil will be found a constant product of the decomposition of glucosides in the manner alluded to. I hope to refer again to this when I shall have been able to look over my former experiments with regianine and fragarine.

TWENTIETH ANNUAL REPORT OF THE COMMITTEE ON INDEXING CHEMICAL LITERATURE.*

THE Committee on Indexing Chemical Literature, appointed by your body in 1882, respectfully presents to the Chemical Section its Twentieth Annual Report, covering the ten months ending June 1, 1902.

Works Published.

"A Bibliography of the Analytical Chemistry of Manganese, 1785—1900." By Henry P. Talbot and John W. Brown. City of Washington, published by the Smithsonian Institution, 1902. 8vo. viii. + 124 pp.

Smithsonian Miscellaneous Collections, Vol. XLI. (No. 1313).

"Index to the Literature of the Spectroscope" (1887—1900, both dates inclusive), [continuation of the previous index by the same author published in 1888]. By Alfred Tuckerman. Washington City, published by the Smithsonian Institution, 1902. 8vo. iii. + 373 pp.

Smithsonian Miscellaneous Collections, Vol. XLI. (No. 1312).

"Chemical Societies of the Nineteenth Century." By Henry Carrington Bolton. City of Washington, published by the Smithsonian Institution, 1902. 8vo. 15 pp.

Smithsonian Miscellaneous Collections, Vol. XLI. (No. 1314).

This contains a list of the serials published by the Societies, fifty-six in number, statistics of membership for 1900, &c.

"On a System of Indexing Chemical Literature," adopted by the Classification Division of the U.S. Patent Office. By Edwin A. Hill. *J. Am. Chem. Soc.*, xxii., No. 8; also *CHEM. NEWS*, vol. lxxxiv., 202 et seq. Oct.—Nov., 1901.

"A Bibliography of Photography," by Miss Adelaide M. Chase, was begun in the February number of the *Photo Era*, published at Boston. It is confined to literature in English, and does not include articles in photographic and chemical journals.

Notes of Foreign Bibliographies.

Krupsky, A. K. "Russkaya chast Khimicheskoy Bibliographii." S. Petersburg. 1900. 62 pp., 4to.

This is a reprint by the Imperial Academy of Sciences, St. Petersburg, of the Russian titles in Bolton's "Selected Bibliography of Chemistry" (Vols. I.—III.).

W. R. Whitney and J. E. Ober. "Index to the Literature of Colloids." *J. Am. Chem. Soc.*, vol. xxiii., p. 842. (Nov., 1901).

Glaser, Langfurth, und Voigtländer. "Sammelkatalog der in Hamburger öffentlichen Bibliotheken vorhandenen Litteratur aus der Chemie und aus verwandten Wissenschaften." Hamburg, 1901. 108 pp. 8vo.

* Advance proofs from *Proceedings of the American Association for the Advancement of Science*, 1902, vol. II.

Garçon, Jules. "Répertoire général ou Dictionnaire méthodique de bibliographie des industries tinctoriales et des industries annexes." Paris, 1900-1901. 3 vols., roy. 8vo. 1978 pages in the three volumes.

This completes the work noticed in the Eighteenth Annual Report.

Works in Progress.

The MS. of an "Index to the Literature of Thorium," by Cavalier H. Joüet, Ph.D., of Columbia University, New York, has been examined by each member of your committee, and recommended for publication to the Smithsonian Institution. It is now being prepared for printing.

Mr. Benton Dales, of Cornell University, has in preparation "An Index to the Literature of the Yttrium Group of the Rare Earths."

Reports of progress have been received from Messrs. Frank R. Fraprie and H. Carrington Bolton.

It is the sad duty of the committee to record the death of one of its original members, Professor Albert R. Leeds, Ph.D., long Professor of Chemistry in the Stevens Institute of Technology, Hoboken, N.J. His contributions to chemical bibliography include "Indexes to the Literature of Ozone," and of "Peroxide of Hydrogen."

It is gratifying to note the increasing and continued interest in bibliography on all sides, and the committee stands ready to encourage the movement in chemistry by practical assistance to those desirous of contributing to the now considerable list of indexes. Address correspondence to the Chairman, at the Cosmos Club, Washington, D.C.

Committee:—

H. CARRINGTON BOLTON (in Europe),
F. W. CLARKE,
A. B. PRESCOTT,
ALFRED TUCKERMAN,
H. W. WILEY.

APPENDIX.

Bibliographies Named in Reports, I-XX., 1883-1902.

The following list includes only those bibliographies that have been published under the auspices of the committee, or independently, as monographs and separates.

Copies may be obtained, so far as available, on application to the Society or Institution issuing them, or in some cases by addressing the authors.

Aceto-Acetic Ester, Bibliography of. By Paul H. Seymour. Smithsonian Miscellaneous Collections, No. 970. Washington, 1894. 8vo.

Carbides, Review and Bibliography of the Metallic. By J. A. Mathews. Smithsonian Miscellaneous Collections, No. 1090. City of Washington, 1898. Pp. 32, 8vo.

Cerium and Lanthanum, Indexes to the Literature of. By W. H. Magee. Smithsonian Miscellaneous Collections, No. 971. Washington, 1895. Pp. 43, 8vo.

Chemistry, A Select Bibliography of; 1492-1892. By Henry Carrington Bolton. Smithsonian Miscellaneous Collections, Nos. 850. Washington, 1893. Pp. 1212, 8vo.

First Supplement, S. M. C., No. 1170, 1899.

Section VIII., Academic Dissertations, S. M. C., No. 1253, 1901.

Columbium, Index to the Literature of; 1801-1887. By Frank W. Traphagen. Smithsonian Miscellaneous Collections, No. 663. Washington, 1888. Pp. iv.+27, 8vo.

Didymium, Index to the Literature of; 1842-1893. By A. C. Langmuir. Smithsonian Miscellaneous Collections, No. 972. Washington, 1895. Pp. 20, 8vo.

Explosives, Index to the Literature of; Part I. By Charles E. Munroe, Baltimore, 1886. Pp. 42, 8vo. Part II., Baltimore, 1893. Pp. 43-195, 8vo.

Electrolysis, Index to the Literature of; 1784-1880. By

W. Walter Webb. Annals of New York Academy of Sciences, vol. ii., No. 10, 1882. Pp. 44, 8vo.

N.B.—This has been translated into French by Donato Tommasi, Paris, 1889.

Heat, Dictionary of the Action of Heat upon certain Metallic Salts, including an Index to the principal literature upon the subject. Compiled and arranged by J. W. Baird; contributed by A. B. Prescott, New York, 1884. Pp. 70, 8vo.

Light, Chemical Influence of, A Bibliography of. Alfred Tuckerman. Smithsonian Miscellaneous Collections, No. 785. Washington, 1891. Pp. 22, 8vo.

Manganese, Index to the Literature of; 1596-1874. By H. Carrington Bolton, Annals of the Lyceum of Natural History, New York. Vol. xi., November, 1875. Pp. 44, 8vo.

Manganese, Analytical Chemistry of; A Bibliography of the. By Henry P. Talbot and John W. Brown. Smithsonian Miscellaneous Collections, No. 1313. City of Washington, 1902. Pp. 8vo.

Morphine, Chemical Bibliography of; 1875-1897. By H. Brown. Pharmaceutical Archives. Vol. i., No. 3. No date, no place. Pp. 60, 8vo.

Ozone, Index to the Literature of; 1785-1879. By Albert R. Leeds, Annals of the New York Academy of Sciences. Vol. i., No. 12, 1880. Pp. 32, 8vo.

Ozone, Index to the Literature of; 1879-1883. Accompanied by an Historical, Critical Résumé of the Progress of Discovery since 1879. By Albert R. Leeds. Annals N. Y. Academy of Sciences. Vol. iii., p. 137. 1884. Pp. 16, 8vo.

Peroxide of Hydrogen, Index to the Literature of; 1818-1878. By Albert R. Leeds. Annals of the New York Academy of Sciences. Vol. i., No. 13, 1880. Pp. 11, 8vo.

Peroxide of Hydrogen, Index to the Literature of; 1879-1883. By Albert R. Leeds. Annals of New York Academy of Sciences. Vol. iii., p. 153, 1884. Pp. 3, 8vo.

Platinum Group, A Bibliography of the Metals of the. Platinum, Palladium, Iridium, Rhodium, Osmium, Ruthenium, 1748-1896. By James Lewis Howe. Smithsonian Miscellaneous Collections, No. 1084. City of Washington, 1897. Pp. 318, 8vo.

Spectroscopy, Index to the Literature of. By Alfred Tuckermann. Smithsonian Miscellaneous Collections. No. 658. Washington, 1888. Pp. x.+423, 8vo.

The same, 1887-1900, both dates inclusive. S. M. C., No. 1312. Washington City, 1902. Pp. iii.+373, 8vo.

Starch Sugar, Bibliography of. By Edw. J. Hallock. Appendix E to Report on Glucose prepared by the National Academy of Sciences, in response to a request made by the Commissioner of Internal Revenue. U.S. Internal Revenue, Washington, D.C., 1884. Pp. 44, 8vo.

Tannins, Index to the Literature of. By Henry Trimble. The Tannins. Philadelphia, 1892. Vol. i., Appendix. Vol. ii., Philadelphia, 1894.

Thallium, Index to the Literature of. By Martha Doan. Smithsonian Miscellaneous Collections, vol. xli., No. 1171. Washington, 1899. Pp. 26.

Thermodynamics, Index to the Literature of. By Alfred Tuckerman. Smithsonian Miscellaneous Collections. No. 741. Washington, 1890. Pp. vi.+329, 8vo.

Thorium, Index to the Literature of. By Cavalier H. Joüet. Smithsonian Miscellaneous Collections, No. —. [In press].

Titanium, Index to the Literature of; 1783-1876. By Edw. J. Hallock. Annals of the New York Academy of Sciences. Vol. i., Nos. 2 and 3, 1877. Pp. 22, 8vo.

Uranium, an Index to the Literature of; 1789-1885. By H. Carrington Bolton. Smithsonian Report for 1885. Washington, 1885. Pp. 36, 8vo.

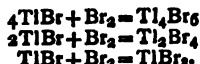
Vanadium, Index to the Literature of. By G. Jewett Rockwell. Annals of the New York Academy of Sciences, vol. i., No. 5, 1877. Pp. 32, 8vo.

Zirconium, Index to the Literature of. By A. C. Langmuir and Charles Baskerville. Smithsonian Miscellaneous Collections, No. 1173. City of Washington, 1899. 29 pp. 8vo.

THE ACTION OF BROMINE ON THALLOUS CHLORIDE IN THE PRESENCE OF WATER. COMPOUNDS OF THE TYPE Tl_2X_4 .

By V. THOMAS.

I. The per-salts of thallium, such as the tribromide, $TlBr_3$, when treated with appropriate quantities of thallous bromide, give rise to intermediate salts, Tl_2Br_4 and Tl_4Br_6 . The problem of the formation of these intermediate salts appears as if it ought to be solved in a much more simple manner by the successive addition of bromine to thallous bromide. The experiment is easy to carry out, and the results seem *a priori* to leave no doubt as to the regular fixation of the bromine on the proto-salt. It is only necessary to take a few elementary precautions to obtain, according as we wish, either Tl_4Br_6 or Tl_2Br_4 . The reactions are then easy to express:—



This regular fixation of bromine on the thallous chloride is, however, only apparent. In fact, it results from very complex reactions which are effected in the body of the solution.

The bromisation of thallous bromide does not lend itself very well to the study of these reactions. These reactions are, on the other hand, very distinctly observed in the fixation of bromine on the chloride $TlCl$. By admitting the fact of the regular fixation of the halogen we ought to obtain as product of the reaction $Tl_4Cl_6Br_2$ and $Tl_2Cl_4Br_2$. Experience shows that we generally obtain bodies altogether different; in particular, the addition of one atom of bromine to two molecules of the chloride leads to the chlorobromide, $Tl_4Cl_3Br_3$. Everything seems to pass as if, in the cold, there was a displacement of a part of the chlorine by the bromine and a simultaneous fixation of the bromine on the product formed.*

Now, under the conditions of the experiments the displacement of the chlorine by the bromine appears to be so singular as to be inadmissible. We think that all the facts we shall state later on agree very well with one of the following hypotheses:—

1. The addition of a quantity of bromine, however small it may be, to thallous chloride gives rise to a thallic chlorobromide of the normal addition $TlClBr$.†

This latter, in the presence of an excess of thallous chloride, is reduced with the formation of chlorobromides belonging to the types Tl_4X_6 and Tl_2X_4 . The reduction to the state of intermediate chlorobromides is never complete. All these chlorobromides are, in fact, dissociated on contact with water, and can only exist in the presence of a variable quantity of the thallic salt.

2. The bromine becomes fixed regularly on the thallous chloride, giving normal derivatives of addition, such as $Tl_4Cl_6Br_2$ and $Tl_2Cl_4Br_2$, but these being dissociable on contact with water, decompose immediately into per-salts and less chlorobromised salts.

II. If to a small quantity of thallous chloride suspended in water we add an excess of bromine the thallous chloride is rapidly dissolved with an accompanying rise of tem-

perature. The solution obtained by evaporation in the air rapidly takes a syrupy consistence, and at the same time it deposits a small quantity of black oxide, Tl_2O_3 , the product of a secondary reaction, the hydrolysis of the chlorobromide formed. At this moment the solution contains a hydrated thallic chlorobromide, $TlClBr_2$, which is very unstable. If we continue the evaporation *in vacuo* over sulphuric acid we obtain a product of decomposition of this chlorobromide of the type Tl_2X_4 with the composition $Tl_2Cl_3Br_4$. Finally, this latter in the presence of water decomposes in the same way as the bromide of thallium, giving rise to a thallic compound, which dissolves, and to a compound of the type Tl_2X_3 , which is precipitated on account of its slight solubility.

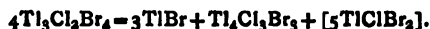
By proceeding in the inverse manner, that is to say, by adding to equal quantities of thallous chloride increasing quantities of bromine, we pass from the compounds of the type TlX to the compounds Tl_2X_3 , Tl_2X_4 , and Tl_2X_6 .

III. By adding an excess of bromine to thallous chloride and evaporating the solution thus obtained *in vacuo*, we can easily obtain the chlorobromide $Tl_4Cl_3Br_3$. (For the details of this preparation see *Bull. Soc. Scient. et Méd. de l'Ouest.*, vol. x., p. 11). It occurs in small transparent prisms, apparently orthorhombic, of a sulphur-yellow colour, and which appear to become tarnished after prolonged exposure to the air.

On contact with water it decomposes according to the equation:—



In every case we obtain, at the same time as the chlorobromide $Tl_4Cl_3Br_3$, a small quantity of thallous bromide; this latter apparently being formed by a decomposition which can be represented by the equation (*Bull. Soc. Scient. et Méd. de l'Ouest.*, vol. x., p. 12—14):—



The hydrochloric acid in solution reacts on the $Tl_4Cl_3Br_3$ in the same way as water; in any case the chlorobromide formed, Tl_2X_3 , is again submitted to the action of the acid, and appears to be brought back to the type TlX . Hydrobromic acid behaves in a similar manner, but the reaction is a little more complicated, the hydrochloric acid being displaced by the hydrobromic acid, at least to some extent.

The oxygenated acids, sulphuric acid, and nitric acid, for example, set a large quantity of the halogen at liberty, especially when heated.

Under the action of heat, at about 150° , the colour of this last salt becomes rather deeper, then at 165° it melts, giving a yellow liquid, and undergoing slight decomposition. At a higher temperature the decomposition is much more rapid, large quantities of bromine are given off, and if we have taken care not to heat too strongly, but only just up to 360° , the residue consists of an orange-coloured mass approaching red, formed of small flakes, very probably belonging to the type Tl_4X_6 .—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 11.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 7th inst., The Duke of Northumberland, K.G., President, in the Chair. Mrs. Baily, Miss L. M. Burnett, the Rt. Hon. Sir Ernest Cassel, K.C.M.G., Lady Kelvin, Miss F. A. Musgrave, Mrs. E. Otter, and Mr. E. Schweich were elected members.

Sewage Disposal.—On the advice of Professor Henry Robinson, M.Inst.C.E., the District Council of Malling, Kent, have decided to lay down the latest type of bacteria beds for Aylesford and Burham, and have sealed a contract for the supply of Candy Automatic R-evolving Sprinklers, and Patent Inter-mitters, for distributing the sewage over the surface of the beds.

* It must be well understood that we do not here advance any hypothesis as to the order in which these two operations might take place.

† This chlorobromide can be easily prepared, and will be described later on.

RECENT DEVELOPMENTS IN COLOURING-MATTERS.*

By Geheimrath Professor OTTO N. WITT, Ph.D. F.C.S.,
of Berlin.

THE love of colour is innate in the human mind, and this alone, if nothing else, would be sufficient to account for the interest with which the coal-tar colour industry has met from its beginning. In England especially its progress has been watched with great attention, and only two days ago the Vice-Patron of this Institution, His Royal Highness the Prince of Wales, has shown by some remarks, made in his Opening Address of the New Technical Institute at Bushey, that the interest taken in this subject has in no way abated.

Artificial colouring-matters have formed so often the subject of more or less popular lectures, and this subject has been treated with such ability by eminent scientists, that it becomes difficult to show this domain of chemistry in a light new and interesting to an audience such as I have to-day the honour to address. Many years ago you have seen in this room the early achievements of the newly-created industry, marvellous for their beauty and brilliancy. Later on the progress of this industry has been duly recorded. More recently still it has become the custom in this country to view colour-making not so much from its chemical or industrial side, as from the standpoint of the national economist, who contemplates the values produced by industrial enterprise, and investigates the reasons why these values should be unevenly distributed amongst the different nations, striving side by side for progress and engaged in friendly, yet none the less eager competition.

I may say at once, that I have no intention to treat my subject from either of these points of view. I take it for granted, that everybody is acquainted with the marvellous variety and brilliancy of artificial dye-stuffs, and I am too much of a chemist and too little of an economist to offer any original or valuable view about that side of the question which I have just mentioned. But I shall make an attempt to trace in this lecture the influence of the development of theoretical chemistry on the progress of the colour industry. If in so doing I should refer now and then to theoretical points without being able to explain them in detail, I hope to be forgiven.

In beginning this lecture allow me briefly to refer to the history of it.

When I received from Sir William Crookes the flattering invitation to speak before you this evening, my thoughts naturally wandered back to some recollections in connection with this Institution. I remembered vividly several brilliant lectures to which I had the privilege of listening in this room, where the spirits of Davy and Faraday, of Graham and Huxley, of Würtz and of my immortal friend A. W. von Hofmann seem still to be hovering. I felt loth to raise my own voice in such hallowed precincts. But then I also remembered an almost forgotten episode in my own life, which I ask your permission to tell.

I remembered, that almost exactly five-and-twenty years before receiving this invitation, I, then a very young chemist, had read before the Chemical Society of London a paper containing a then somewhat daring speculation on the connection of the constitution of colouring-matters with their properties, a paper which the Publication Committee refused to print. A lively discussion followed, which was wound up by some encouraging remarks from the President, the late Mr. De la Rue. He said that he hoped that this speculative paper would prove useful in clearing up the complicated domain of colouring-matters, and that perhaps on some future occasion I should be in a position to place before the world, in a *Royal Institution lecture*, the results which had been obtained by its help.

This strange reminiscence, coupled with the curious

fact that Mr. De la Rue's prophetic words were fulfilled just when the period commonly assigned to a Jubilee had elapsed, gave me the courage to accept Sir William's kind invitation. For though I have done comparatively little towards the increase of our knowledge of colouring-matters, the five-and-twenty years past have sufficed to shed a brilliant light on what Mr. De la Rue could then justly call a very imperfectly known domain of chemistry, and innumerable facts brought to light during this period by a whole army of assiduous workers are now by common assent being classified under a theory which is neither more nor less than the suggestions contained in that rejected paper of mine, which I had fortunately published in another journal.

I may add, that I have been guilty in later times of another theory, which refers to the domain of dyeing, and which has still many opponents. This theory is the direct outcome of the theory of colouring-matters, and may be illustrated by some simple, yet striking experiments, some of which I intend to show you.

A fundamental question in the chemistry of dye-stuffs, and one not at all easy to answer, is this: "What is a dye-stuff?" Clearly it is something totally different from a substance only endowed with the power of selective absorption of light, a power which causes it to appear coloured. We know now that there are more substances in creation which possess this power than bodies which lack it. In this very room we have learned that the air itself, through which the solar rays penetrate on to the surface of the earth, is blue and not colourless, as we used to think. But even if we leave out of the question such faintly coloured substances as air and water, if we restrict our consideration to compounds endowed with a very intense power of selective absorption and at the same time soluble in the water which we employ for preparing our dye-baths, we do not arrive yet at the true definition of the dye-stuff. Cupric salts, soluble chromates, and many other intensely-coloured bodies are no dye-stuffs, as may be easily shown by experiment. Yet these compounds penetrate into the interior of textile fibres which are immersed into their solution. They must do so, according to the laws of Osmose so ably expounded by Thomas Graham, because they are crystalloids and the fibres are invariably colloids.

We know now that the laws of Osmose are identical with the laws governing solution, and that crystalloids are able to wander into the interior of colloids because they are soluble in their substance. Osmotic processes may be observed between two liquids which cannot be mixed with each other, just as well as between a liquid and a colloid immersed into it. Consequently we are justified in assuming that the same powers are at work in both cases.

In my first experiment (Exp. I.)* I intend to show you that a crystalloid, dissolved in some liquid such as water, and brought into contact with another liquid, not miscible with the first, such as ether, will either remain indifferent to the ether altogether, or it will leave the water and wander into the ether, or it will be distributed according to a certain ratio between the two solvents. In this latter case we have reason to believe that a constant interchange of molecules takes place between the two solutions. Clearly, this will only happen if there exists no great difference in the solubilities of the crystalloid in water and in ether. In that case the water will continually abstract nearly as many molecules of the crystalloid from the ether as the latter will take up from the water, and thus an equilibrium will be reached. If, on the other side, there is a great dissimilarity in the solubility of the crystalloid in the two solvents, then this process of mutual interchange will become so one-sided that it practically amounts to

* Details of experiment: An aqueous solution of magenta does not yield its colouring-matter to ether; indophenol, on the contrary, is entirely taken up by ether. The dye-stuff, which is partly taken up by ether, is also a member of the indophenol group, the constitution of which is not yet fully established.

* A Lecture delivered at the Royal Institution of Great Britain, Friday, March 21, 1902.

the absorption of the whole of the crystalloid by one of the solvents.

My second experiment (Exp. II.)* is a more striking illustration of these fundamental facts. If we mix together two coloured solutions, one an aqueous one of a substance much more soluble in ether than in water, and the other an ethereal one of a substance more soluble in water than in ether, then the two solutions, on shaking, change colour, and their shades are reversed.

According to my theory, the process of dyeing, considered so problematical by many experts in this ancient and useful art, is strictly analogous with this wandering of molecules governed by the laws of solution, which we can so easily observe and control in operating with two non-miscible liquid solvents.

In my next experiment (Exp. III.)† we see that a dye-stuff wanders from the bath on to the fibre in much the same way as it wandered from water into ether. And if the fibre be previously dyed with a colouring matter little soluble in its substance, then this may be expelled and replaced by another of greater solubility. (Exp. IV.)‡

We see now that, in order to become a dye-stuff, a substance must not only be so intensely coloured that it can communicate its own shade to colourless substances holding it in solution; it must not only be soluble in water or any other liquid suitable for preparing a dye-bath; but it must also be soluble, and even much more soluble than in water, in the colloid, which forms the substance of the textile fibre. The finished dyed fabric is nothing more nor less than a solid solution of the dye-stuff in the substance of the fibre, unless there are secondary chemical influences, such as that of the mordants, at work, which change the solution into a suspension by precipitating the dye-stuff after its immigration into the fibre.

This peculiar combination of solubilities is very rarely met with amongst the coloured substances of an anorganic nature. In the vast domain of organic compounds of the aliphatic series we meet with very few dye-stuffs, because its members are mostly colourless, or but very faintly coloured. In the aromatic series, on the contrary, the power of selective absorption of light is so very frequent, that it would be very curious indeed if just that combination of solubilities, which is the making of the dye-stuff, were not of common occurrence. Taking as a basis the universally admitted axiom, that the physical properties of every compound are direct functions of its molecular constitution, we may easily believe that that peculiar combination of solubilities which I have shown to be the characteristic feature of the dye-stuff, would be the result of certain general conditions fulfilled in the constitution of many members of the aromatic group. My theory, proposed five-and-twenty years ago, was nothing else than an attempt to ascertain these general conditions by investigating the constitutional peculiarities of all those dye-stuffs the constitution of which had been fully established in those days.

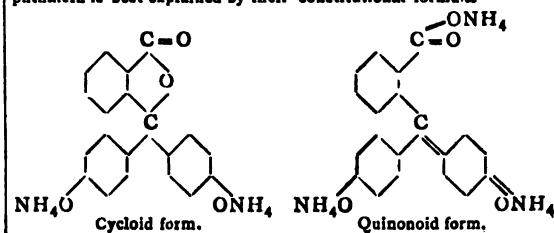
I have no intention to tax your patience by explaining in detail the results of that old investigation. It will be sufficient to summarise them by saying that in the molecule of every colouring matter, the constitution of which has been ascertained to this day (and there are many thousands of them), certain atomic constellations have been observed which seem to be essential, and of which always two must be present. One of these constellations is a group of atoms, which is the cause of the selective absorption of light. This group of atoms I call a *chromophore*. The number of atomic groups endowed with chromophoric properties amounts at present to about two dozen, and is being constantly increased by the progress of chemical research. All the chromophores, however, have that in common, that they are unable to exert their

influence unless they are helped by the presence of another group of atoms, which I call the *auxochromic* group. Very few auxochromic groups are known, and they belong to those which occur most frequently in the whole domain of organic chemistry—the amino group in its various forms, the hydroxyl group occurring in all the phenols, the sulpho- and the carboxyl group. None of these will cause a substance to become a dye-stuff unless this substance also contain a chromophore, but the latter is equally helpless if deprived of the assistance of the auxochromic group. Thus we meet in the molecular world that condition of the necessity of mutual help and assistance between two heterogeneous forms, which we can also trace in Sociology, a fact the establishment of which will no doubt be greeted with satisfaction by the ladies in this audience.

Our ideas on the nature and constitution of those groups which may act as chromophores have of course undergone many changes. Undoubtedly there must exist a law which governs the formation of chromophoric groups, but so far this law has not been definitely established. Some progress has, however, been made towards this end. At first the chromophores which we had gradually collected formed rather a motley crowd, and seemed to have no points in common. At present chemists working in this domain are inclined to attribute a quinoid structure to the great majority of colouring-matters. If this view be correct, then all these substances would be derivatives, not of benzene and its congeners, but of hydrocarbons containing two hydrogen atoms more in their molecule, derived from dihydrobenzene as a prototype. As sometimes it is almost impossible to decide in favour of one view or the other, the convenient hypothesis of tautomerism was resorted to, but in some cases we have been able to establish definitely the quinoid formula. Such is the case with the large and brilliantly-coloured group of dye-stuffs called phthaleins, which, according to modern view, must be considered as quinoid derivatives of benzoylbenzoic acid. The experiments which lead to this conclusion are so striking, that I cannot refrain from producing one of them, which has never been shown yet, though the time at my disposal does not allow its exhaustive discussion from a theoretical point of view. If we dissolve the well-known phenolphthalein in anhydrous ether containing some ammonia, the solution is perfectly colourless, but if we add ordinary water to this solution (Exp. V.), it assumes a beautiful red colouration. This peculiar fact that water alone is sufficient to cause the formation of this colour is perfectly incomprehensible if the old views on the constitution of phthaleins, which are still given in the majority of text-books, be adhered to, but it is exactly what we might expect to happen if we assume that the ammonium salt of phenolphthalein possesses a cycloid constitution in its ethereal solution, and that it is isomerised into the quinonoid form by the addition of water.*

Thus our knowledge of the chemical causes of the physical properties of colouring-matters is continuously developing. Quite lately we have even begun to form definite views about the connection of the chemical constitution of aromatic substances with that peculiar form of selective absorption of light which we call fluorescence,

* The isomerism of the two forms of the ammonium salt of phenolphthalein is best explained by their constitutional formulae—



* The ethereal solution used contained magenta acetate, whilst the aqueous one was prepared with tricalcro-indophenol.

† In Exp. III. wool was dyed with erythrosine in the ordinary way, whilst in Exp. IV. a cotton cloth, previously dyed with patent blue, was treated in a bath of Congo red.

and which has formed, from the physical point of view, the subject of the masterly investigations of Sir Gabriel Stokes. The phenomenon of fluorescence is very frequently met with in dye-stuffs, and in the raw materials used for their manufacture. It can be exhibited in a very striking way with the help of electricity, either by allowing an arrow of electric light to penetrate into the solution of a fluorescent substance or by working a Geissler tube of suitable shape submerged in such a solution (Exp. VI.). The fact that the fluorescence of many substances is chiefly caused by the ultra-violet light, I shall try to demonstrate by the following somewhat delicate experiment: I have here, submerged in a solution of eosine, a Geissler tube, the lower part of which is ground out of a piece of rock-crystal, whilst the upper half is made of glass. When the electric current passes this tube the fluorescence round the quartz part of it is stronger than that in the neighbourhood of the glass, because the latter absorbs a good deal of ultra-violet light, whilst the quartz is almost free from such absorption. (Exp. VII.)

An immense amount of patient work has been accomplished by many chemists in the hope of establishing definite views on the constitution of the azo-colours, that group of dye-stuffs the introduction of which into the colour industry was the direct consequence of our early efforts to cast off empiricism, and to conduct our search for new colouring-matters according to definite scientific principles. Simple and transparent as the constitution of azo-colours appears to be if viewed superficially, yet it offers some problems of extraordinary difficulty, which have not been solved so far. But fortunately these difficulties have in no way interfered with the technical development of this family of dye-stuffs, which has been for a whole quarter of a century one continued and unparalleled series of successes. The process for producing these dye-stuffs is of the greatest simplicity. It consists in pouring together (Exp. VIII.) cold aqueous solutions or suspensions of diazo-compounds and phenols or amines. The dye-stuff is formed at once in a state of absolute purity, and with a yield absolutely theoretical; it need only be collected and dried to form a saleable product. No wonder, then, that these dye-stuffs gradually became the leading ones, and to a great extent superseded the old empirical products which were concocted in many complicated operations, with yields very far from satisfactory. As the number of diazo-compounds and of phenols and amines at our disposal is very large, the number of dye-stuffs which may thus be prepared is quite extraordinary; it has been computed, according to the rules of permutation, 3,159,000 different individual dye-stuffs have thus been proved to be at present easily accessible to our industry. Of these at least 25,000 form the subject of German patent specifications and of corresponding specifications in England, France, the United States, and other countries. Over five hundred are regularly manufactured on the larger scale.

The prolific nature of the azo-colour reaction explains the fact, that in this group we can choose, much better than in any other, substances possessing that ratio of solubilities in water and in the colloid substance of the various textile fibres, which we require. We can produce quite at will, azo-dye-stuffs which dye wool or silk or cotton, which dye slowly or quickly, which will stand soap or acid or alkali. This possibility of adjusting the chemical properties of dye-stuffs with an almost mechanical precision has been the cause of one of the greatest successes of the colour industry, the introduction of what is now known under the name of "substantive dye-stuffs," an expression which means dye stuffs that will dye cotton and other vegetable fibres from a simple aqueous dye-bath without the use of any mordant. The difference of the solvent power of cellulose and of water is for the vast majority of dye-stuffs so small, that the process of dyeing vegetable fibres with these ordinary colouring-matters can only be compared to that case of the joint action of ether and water on some substance soluble in both these solvents, where an almost equal division of

this substance takes place between the two solvents. Such cases exist as you saw in the first experiment. It is amongst the azo-dyes that we have found compounds which are so much more soluble in cellulose than in water, that they readily leave their aqueous solution and take up their abode in the fibre. And we have not only found these dye-stuffs but also the law which governs this most valuable abnormal solubility; it appears in all azo-colours which are prepared with diazo-compounds derived from symmetrical para-diamines. A novel and extremely fertile field for a systematic search for new dye-stuffs was thus opened, a field which has occupied hundreds of busy workers for many years, many of whom carried home a rich reward.

But whilst this field bore its rich harvest, others were by no means neglected. The search for dye-stuffs, which will dye cotton without a mordant, could not make us forget that just those colouring-matters which imperatively demand the use of mordants are those which from times immemorial have been used in preference for the production of fast and lasting shades. The brilliant synthesis of alizarine by Graebe and Liebermann, which made the world ring with admiration early in the seventies, had given us ample proof that the old, and to this day not wholly forgotten axiom, that there are two kinds of dyes—natural ones, which are fast, and artificial ones, which are fugitive—was a preconceived idea, totally devoid of any scientific foundation. The enormous financial success of the alizarine industry formed a tempting invitation to search for other dye-stuffs, which, similar to alizarine, would be endowed with the power of forming almost indestructible lakes with mordants of a sesquioxycidic nature. Here too, like everywhere in science, we have marched for some time on the paths of empiricism, but here too logical deduction has come to our aid in disclosing the laws which govern the formation of lakes. In this case it is not (as in the substantive azo-dyes) the carbonic nucleus which determines the physical properties (*viz.*, the ratio of solubilities) of the dye-stuff, but it is the peculiar position of the auxochromic groups contained in the molecule which governs its chemical properties. We know now that a dye-stuff must contain, in order to be able to form lakes with sesquioxycidic mordants, two hydroxyl groups in juxtaposition. If this condition be fulfilled, the dye-stuff will dye in the same way and with equal fastness as alizarine, even if it be no derivative of anthracene, like the early alizarine dyes; and if these two hydroxyl groups or a suitable equivalent for them be missing, it will lack all power of dyeing mordants, even though derived from anthracene. With this law once established the synthesis of mordant dye-stuffs became a very easy matter, and to-day there is hardly a group of colouring-matters in which there are not some members possessed of this peculiarity, and owing it to the same uniform cause. Still the group of the oxyketones, to which alizarine itself belongs, remains the true home of mordant-dyes, but this group has grown to-day into a very numerous and varied one. Mordant-dyes of every shade are to be found in it, and cotton is no longer the only fibre to which such dyes are applied. It is a fact worthy of notice, that amongst the many dyes of this class which we now possess and the constitution of which is fully established, there is not a small number, the molecule of which contains three, four, five, or even six hydroxyl groups. Yet this increase of auxochromic groups does not influence in the least the behaviour of these dyes to mordants; this is only governed by the two hydroxyl groups in ortho-position, and any other such group introduced into the molecule only changes the shade, not the characteristic chemical properties.

A greater variety still than by the achievements of modern synthetical work will come into this group of mordant-dyes by the progress of the elucidation of the constitution of the natural dye-stuffs occurring in roots, barks, and woods. A good many of them are still unsolved mysteries, but there can be no doubt that they owe, like alizarine, purpurine, and the other madder dye-stuffs,

their property of dyeing metallic mordants to the presence of hydroxyl groups in ortho-position in their molecule.

A very large and varied group of colouring-matters, which for a long time resisted all attempts at unravelling their constitution, are the safranines, curhodines, oxazines, thionines, indulines, and other allied groups. They are now completely understood, and have been recognised as the amino- and oxy-derivatives of certain peculiar substances such as the azines and azonium-bases, the molecule of which possesses a ring-structure. Here no longer carbon atoms only form the closed chain, but nitrogen, oxygen, and even sulphur atoms participate in its structure and bring about the peculiar properties of the compounds. When this fact was at first ascertained it seemed sufficient for the explanation of the behaviour of such compounds as dyes. It was only somewhat later on that we recognised that in these classes of dye-stuffs especially a quinonoid structure is essential.

The greatest and most brilliant success of the chemistry of dye-stuffs is, however, the industrial synthesis of indigo. This offers so many points of general interest that I am sure to meet with your approval if I refer to it in some detail.

The indigo problem is one of the oldest problems of chemistry. When Baeyer took it up more than thirty years ago he found the ground well prepared by others who had worked before him. But his is the merit of having completely elucidated the constitution of this extraordinary product of nature. He and others have also shown various methods for the synthesis or artificial production of indigo. In the laboratories artificial indigo has been known for the last twenty years.

But in this case the scientific synthesis of a natural product proved to be by no means identical with the industrial one. Industrial methods can only enter into competition with nature if they work more economically than nature does. In the case of indigo there seemed to be little hope for fulfilling this condition. The most enthusiastic admirers of the modern synthetical industry could not help seeing that all evidence in our hands went against the probability of the practical synthesis of indigo, and just those who understood most of these things could least of all close their eyes to that fact. It could not be denied that every possible synthesis of indigo, those known as well as those which might still be expected, had to start from some aromatic derivative of benzene, containing one carbonic and one nitrogenous side-chain in ortho-position. Of all the products at our disposal which fulfil that condition, orthonitrotoluene is the most easily accessible. Now, taking it for granted that indigo could be prepared regularly and with good yields from orthonitrotoluene, there still remained that difficulty, that all the toluene produced in the world, even if we suppose that all the other uses to which this hydrocarbon is put at present could be suppressed, would not suffice for the production of the world's consumption of indigo.

If, under such circumstances, the industry of artificial dye-stuffs continued to work at the indigo problem, it did so more for the general interest attached to it, and with a view to securing some of the finer applications of indigo in printing, than in the hope of being able to compete with the natural product in the great consumption of vat dyeing. If, on the other hand, the indigo planters in the far East showed but small apprehension of the danger of which they were occasionally warned, we cannot blame them for it; they had no doubt taken the advice of competent people, and these had told them what was correct according to the knowledge of the time.

The final result has shown all the calculations of experts to be wrong, but in such a way that they, too, can surely not be blamed for the error they committed.

The process by which indigo is at present manufactured on a colossal scale by the Badische Anilin- und Soda-Fabrik in Ludwigshafen on the Rhine, is based on Heumann's synthesis of this most important dye-stuff,

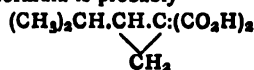
which consists in submitting phenylglycin to a fusion with caustic alkali. Phenylglycin is prepared by the action of monochloroacetic acid upon aniline. The yield of indigo obtained is a poor one, but it can be very much improved if, instead of phenylglycin, we take its orthocarbonic acid. In this we have again the presence of a nitrogenous and a carbonic side-chain in ortho-position. To prepare this acid we should have to start, according to the ordinary rules, from toluene, transforming it by a succession of operations. Thus we come again to toluene as a starting-point, and to the difficulty already explained.

There is, however, one somewhat abnormal process of preparing the same compound from phthalic acid. It consists in converting this into phthalimide, and treating the latter with sodium hypochlorite. By a somewhat complicated reaction, the nature of which need not be explained, one of the carboxyl groups of the phthalic acid is replaced by the amido group, anthranilic acid is formed, and this, if treated with monochloroacetic acid, yields phenylglycin-carbonic acid, which has proved so important for the manufacture of indigo. Now phthalic acid is prepared by a powerful oxidation of naphthalene, and naphthalene again is that constituent of coal-tar which is present in by far the largest quantity.

It is true that the process for transforming naphthalene into phthalic acid, which was the only one known at the time when all these facts were first recognised, gave very bad yields, and was at the same time costly. The whole indigo problem stood thus reduced to the problem of transforming naphthalene cheaply and economically into phthalic acid. This has been accomplished by the Badische Anilin- und Soda-Fabrik by heating naphthalene with fuming sulphuric acid in the presence of mercury salts. Torrents of sulphur dioxide escape, and the whole process can only be carried out properly if the means be given to convert this gas again into fuming sulphuric acid, which may be used again for treating fresh quantities of the hydrocarbon. The new sulphuric acid process of the Badische Anilin- und Soda-Fabrik has thus been of paramount importance for the working out of the indigo problem.

(To be continued).

Reaction of Sodium-malonate of Ethyl on the Dibromides, $C_8H_{12}Br_2$.—V. Ipatief.—In the reaction of sodium-malonate of ethyl on bromide of isopropylethylene, $(CH_3)_2CH \cdot CHBr \cdot CH_2Br$, we obtained the ether of a non-saturated acid, boiling at $122-132^\circ$ under 18 m.m. pressure; saponification gave a non-saturated bibasic acid with the composition $C_8H_{12}O_4$, soluble in benzene and chloroform, but less soluble in water, and fusing at $76-78^\circ$. This acid decolourises permanganate of potash; its formula is probably—



It is distinguished from its isomer, dimethylallylmalonic acid, $(CH_3)_2C \cdot CH \cdot CH_2 \cdot C(CO_2H)_2$, inasmuch as it is soluble in chloroform, and has a soluble salt of calcium and a silver salt more soluble in water. In the reaction of sodium-malonic ether on the dibromide in question, no carbide is formed, but there are traces of acetyltetracarboxylic ether. Thus, the dibromides in which the Br's are primary and secondary, behave with regard to the sodium-malonic ether differently to those in which one of the Br's is tertiary, and the other primary or secondary. With the bromide, $(CH_3)_2C(CH_2Br)_2$, the reaction takes in a different manner; it is necessary to heat for a long time, about 100 hours; a crystallised product is then formed, fusing at $105-105.5^\circ$, soluble in alkalis, from which it is precipitated by acids. This body decolourised permanganate of potash; by bromination it gives a material fusible at $127-128^\circ$; its composition answers to the formula $C_7H_8O_4$. Its examination is not yet complete.—*Journ. Soc. Phys. Chim. R.*, vol. xxii., p. 647.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, Thursday, June 5th, 1902.

Prof. THORPE, C.B., F.R.S., Vice-President, in the Chair.

(Concluded from p. 12).

103. "Optically Active Esters of β -Ketonic and β -Aldshydric Acids. Part II. Menthyl Acetoacetate." By A. LAFWORTH and A. C. O. HANN.

Menthyl acetoacetate may be obtained in small quantity by the action of sodium on menthyl acetate, but is far more easily prepared by heating menthol with ethyl acetoacetate until alcohol ceases to come off. These observations were made before the authors were aware that Cohn had prepared the ester by the latter process (*Monatsh.*, 1900, xxi., 200-204). As Cohn has since notified (*Ber.*, 1900, xxxiii., 734) that he had abandoned the work, with his concurrence the authors have resumed the study of this substance and its derivatives.

The compound may be obtained in large, lustrous, probably monoclinic prisms which the authors believe to represent the ketonic form. Its rotation in 2 per cent solution in benzene is initially about $[\alpha]_D = -61-62^\circ$, but slowly increases to about -68° , owing doubtless to partial conversion into the enolic form; similar changes are observed when it is dissolved in chloroform, light petroleum, and dry ethyl acetate. The duration of the change varies greatly with the purity of the solvent, as the compound is exceedingly sensitive to catalytic agents. As usual, all bases accelerate the change, and the mutarotation, unlike that of camphorquinonehydrazone and of nitrocamphor, is accelerated by traces of acids also. Equilibrium between the two forms appears to be attained almost at once in alcohol, practically no alteration of rotation with time being noticeable.

The copper derivative of the ester crystallises from alcohol in green prisms which contain alcohol; these rapidly become opaque, and finally have the melting-point $117-118^\circ$.

The ester readily affords a well-defined semicarbaside, $\text{NH}_2\cdot\text{CO}\cdot\text{N}_2\text{H}_2\cdot\text{CMe}\cdot\text{CH}\cdot\text{CO}_2\text{C}_{10}\text{H}_{19}$, flat needles, m. p. $143-144^\circ$; in benzene $[\alpha]_D = -56.1^\circ$. The p-nitrophenylhydrazide, $\text{C}_6\text{H}_4\text{NO}_2\cdot\text{N}_2\text{H}_2\cdot\text{H}\cdot\text{CMe}\cdot\text{CH}\cdot\text{CO}_2\cdot\text{C}_{10}\text{H}_{19}$, brilliant prisms, m. p. $105-106^\circ$; in benzene $[\alpha]_D = -42.5^\circ$.

Menthyl β -aminocrotonate, $\text{NH}_2\cdot\text{CMe}\cdot\text{CH}\cdot\text{CO}_2\text{C}_{10}\text{H}_{19}$, prepared by the action of gaseous ammonia; forms large, transparent plates, m. p. $88-89^\circ$; in benzene $[\alpha]_D = -105.2^\circ$. Menthyl β -phenylaminocrotonate, $\text{NHPh}\cdot\text{CMe}\cdot\text{CH}\cdot\text{CO}_2\text{C}_{10}\text{H}_{19}$, crystallises in flat, rectangular plates, m. p. $85-86^\circ$; it has $[\alpha]_D = -98.2^\circ$. Menthyl benzylaminocrotonate, —



crystallises in flat needles, m. p. $85-86^\circ$; in benzene $[\alpha]_D = -59.8^\circ$.

Menthyl β -benzoylacetate, $\text{CHBzAc}\cdot\text{CO}_2\text{C}_{10}\text{H}_{19}$, is an oil; in benzene $[\alpha]_D = -44.3^\circ$; its copper derivative forms slender needles, is sparingly soluble in most media, and melts at about 230° .

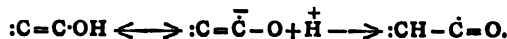
Neither the oxime, the isonitroso-, nor bromo-derivatives of the ester have yet been obtained in a pure condition.

The ester condenses readily with aldehydes, forming, in some cases, well crystallised compounds. Unlike ethyl acetoacetate, when condensed with benzaldehyde at the ordinary temperature by the aid of piperidine, it affords, for the most part, a monobenzylidene compound.

104. "The Mechanism of Simple Desmotropic Change." By A. LAFWORTH and A. C. O. HANN.

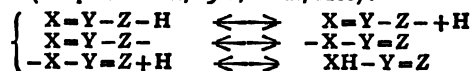
Brühl has advanced the hypothesis (*Ber.*, 1899, xxxii., 2329) that enols (which are weak acids and electrolytes)

are transformed into the isodynamic ketones, by a process which involves initial ionisation into hydrogen ions and organic ions or "residues," which may re-unite in two ways, so that finally the ketone is produced in this manner:—



He found, as he anticipated, that the velocity of transformation of enolic ethyl mesityloxideoxalate in solution into the ketonic form increases roughly with the dielectric constant of the medium.

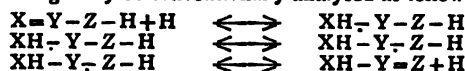
Although the principle here introduced appears quite reasonable, and more capable of experimental and theoretical development than any other yet suggested, it is clearly incomplete, since ketones, although apparently non-conductors, are known to exist in equilibrium with their conducting enolic forms, and the velocity of transformation must be far slower than the ionisation velocity. Since change of internal structure is necessary, and isomerisation appears to increase with ionisation, the authors think that Brühl's suggestion would lead to the following view of the processes involved in a keto-enol transformation (compare *Trans.*, 1901, lxxix., 1266):—



Here it appears necessary to assume that ketones themselves are very slightly ionised, which is consistent with the views of Thiele, Vorländer, and others. According to this view, bases should accelerate and acids retard the change, and the state of equilibrium should be independent of the catalytic agent, but dependent, not only on the two dissociation constants, but also on the velocities indicated in the second line. It is, in fact, perfectly consistent with the properties of certain isodynamic pairs, for example, nitro- and isonitro-camphor (Lowry, *Trans.*, 1899, lxxv., 221) and camphorquinonehydrazones.

However, the complete difference in behaviour of camphorquinonehydrazones and menthyl acetoacetate toward acids indicates that there are at least two classes of substances, and that more than one type of process may go on.

To account for the action of acids in accelerating the speed of migration of a hydrogen atom, in certain cases, on a principle similar to that proposed by Brühl, it seems necessary to assume that the compounds may react as bases. Since, as before, a change of structure is involved, the change may be conventionally analysed as follows:—



where the dot indicates the direction in which the "free affinity" of Y is temporarily disposed, a convention easily understood by reference to models.

The process thus represented requires an acceleration of the change by acids, and a final state of equilibrium independent of their presence.

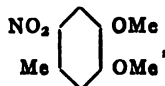
Clearly, a compound might react in either or possibly in both ways, according to the conditions. In nearly all cases observed, however, the first process would appear to go on, the latter being somewhat exceptional, at least as an important factor. Obviously, the state of equilibrium must be the same when attained by either process.

105. "Trimethylbrazilone." By W. H. PERKIN, jun.

During the course of a long series of experiments on the constitution of brazilin, it was shown (W. H. Perkin and A. W. Gilbody, *Proc.*, 1899, xv., 27) that trimethylbraziline, $\text{HO}\cdot\text{C}_{16}\text{H}_{10}\text{O}(\text{OMe})_3$, on oxidation with chromic acid, yields trimethylbrazilone, $\text{HO}\cdot\text{C}_{16}\text{H}_9\text{O}_2(\text{OMe})_3$. It was also stated that nitric acid converts trimethylbrazilone into a substance crystallising in yellow needles, which dissolve in alkalis, forming an intense purple solution; this, on standing, yields besides *p*-methoxybenzoic acid $\text{MeO}\cdot\text{C}_6\text{H}_4(\text{OH})\cdot\text{CO}_2\text{H}$, two neutral substances

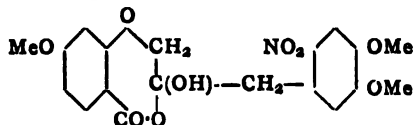
melting at 118° and 206° respectively. Since the yellow substance did not give the usual qualitative tests for nitrogen, it was at first thought that it did not contain this element; subsequent quantitative experiments, however, demonstrated the presence of nitrogen, and showed that the substance has the formula $C_{19}H_{19}O_9N$. It is proposed to name it *nitrohydroxydihydrotrimethylbrazilone*.

The two neutral substances having the melting-points 118° and 206° are probably isomeric, and have the formula $C_9H_{11}O_4N$, and as they each contain two methoxy-groups, they are apparently nitrodimethoxymethylbenzenes, the former (m. p. 118°) being probably identical with the compound—



which has already been prepared by H. Cousin (*Ann. Chim. Phys.*, 1898, [7], xlii., 480), and which melts at 117° . There is, however, a possibility that the very sparingly soluble substance of melting-point 206° has a higher molecular weight such as that represented by the formula $(C_9H_{10}NO_4)_2$.

When nitrohydroxydihydrotrimethylbrazilone is oxidised with permanganate, it yields 2-carboxy-5-methoxyphenoxyacetic acid, and since it also yields an acetyl compound, it is probably a lactone, having the formula—



or some formula closely allied to it.

The author has not yet concluded his experiments on this interesting nitro-compound, but finds it necessary to publish this short note because of a recent publication of Bollina, Kostanecki, and Tambor (*Ber.*, 1902, xxxv., 1675), which contains a description of some experiments on this compound.

The author hopes that these chemists will allow him, for a short time longer, the uninterrupted investigation of this substance.

PHYSICAL SOCIETY.

Ordinary Meeting, June 20th, 1902.

Prof. S. P. THOMPSON, President, in the Chair.

MR. G. F. HERBERT-SMITH exhibited the Three-Circle Goniometer recently constructed for the British Museum from his designs.

In this form of goniometer the advantages of the earlier forms are combined; as with the two-circle or theodolite goniometer, a crystal is only once adjusted during the whole of the observations, and as with the one-circle goniometer observations are made in zones, and full advantage may be taken of the zonal characters of crystals and of the simple formulæ depending thereon. The instrument is an improved form of a similar one designed by the exhibitor in 1899. In place of the usual telescope and collimator an auto-collimating telescope is employed, and a prism is placed before the object-glass so that the line of reference is horizontal and at right angles to the axis of the telescope. In this way it is possible to obtain a reflection from a face parallel to, and on the side of, the crystal towards the vertical circle, and measurements may be made in a zone across the end of the crystal by rotation of the horizontal circle only.

Prof. EVERETT asked whether the reflection was at normal incidence. If so, was not the light very much enfeebled?

Mr. APPLEYARD drew attention to the method of reading

the scales by micrometer eye-pieces, thus saving the use of verniers.

Prof. PERRY suggested that the instrument should be made of nickel-steel alloy of low coefficient of expansion. This alloy is now manufactured in England, and is quite cheap.

Mr. HERBERT-SMITH, in reply to Prof. Everett, said that the image obtained at normal incidence was faint, but it was much more distinct than images obtained with other angles of incidence.

A paper "On the Heat Evolved or Absorbed when a Liquid is brought in contact with a finely-divided Solid," was read by Mr. G. J. PARKS.

Pouillet discovered the fact that when a powder is put into a liquid which does not exert any solvent or chemical action upon it, there is, in general, a rise of temperature. The objects of the present investigation were to obtain a relation between the quantity of heat evolved and the area of the surface exposed, to find the rate of variation of heat evolved with temperature, and to apply to the results the laws of thermodynamics. In making any experiment on the Pouillet effect it is essential that the powder should be perfectly dry, and that it should be at exactly the same temperature as the liquid. The precipitated silica, sand, or other substance to be experimented upon was heated to a dull red heat and placed in a bulb whilst still hot. The air was then exhausted, and the bulb sealed. In making an experiment the bulb containing the powder was broken under the surface of the water in a calorimeter, and the rise of temperature noted. The average diameter of the grains of powder used was obtained by measurement with a microscope, and on the assumption that the grains were spherical the surface of the mass of the powder used was calculated. From the results of his experiments the author states that when silica, sand, or glass is brought into contact with water at approximately constant temperature, the heat evolved is proportional to the area of the surface exposed by the solid, and the amount of heat developed per square centimetre is approximately 0.00105 calorie when the temperature is near 7° C. Assuming that the phenomenon of Pouillet is reversible, and that it is due to a pressure at the surface of the powder, the author has, by the application of the laws of thermodynamics and the results of his experiments, arrived at the conclusion that at 7° C. the surface-pressure of water and silica diminishes at the rate of 157 dynes per centimetre for an increase of temperature of 1° C. Experiments made at different temperatures indicate that the heat evolved is roughly proportional to the absolute temperature. Experiments were also made which showed a fall of temperature on putting a finely divided solid into mercury.

Prof. EVERETT said the research was a valuable contribution to our knowledge of the Pouillet effect. The results were consistent in showing a generation of heat to the amount of about one-thousandth of a therm per sq. cm. of increase of surface of contact between the water and the silica. He thought this heat must be due to diminution of volume in the water-film; for extension of area of a liquid film tended to produce cold. Cold was in fact produced by enlargement of the surface of mercury in the concluding experiments.

Mr. J. MACFARLANE GRAY said that the paper was to him peculiarly interesting, because satisfactory explanations of the phenomena described could be obtained by substituting the universal dynamic pressure of a corpuscular ether for the unthinkable universal attraction of matter. He gave an example to illustrate what he calls the metafilm at a surface, and pointed out that it is necessary to consider both the matter-volume and the meta-volume of bodies to get at the explanation of physical phenomena. In the author's experiments there is a diminution of meta-volume, and the ether produces heat equal to the product of the ether pressure and the volume of the cancelled metafilm, just as the heat of evaporation is reproduced when steam is condensed. The experi-

ments were to him not a surprise, but a confirmation of his views.

Mr. R. S. WHIPPLE said that the Pouillet effect could be shown by covering the bulb of a thermometer with muslin and putting it into a steriliser. On taking it out and immediately placing it in the mouth, there is a considerable rise of temperature. He had tried covering the bulb with dry muslin, and obtained a rise of 2° C. by putting the instrument into water.

Mr. J. H. GARDINER drew attention to the necessity of heating the powdered silica to red-heat in order to get rid of moisture. If possible, the silica should be heated in the actual tube and sealed up whilst hot.

A paper by Prof. R. W. WOOD, "*On a Remarkable Case of Uneven Distribution of Light in a Diffraction Grating Spectrum*," was read by the SECRETARY.

It is a well-known fact that in the spectra formed by diffraction-gratings the light is unevenly distributed, that is the total light in any one spectrum will not re-combine to form white light. The author has been examining a most remarkable grating in which the drop from maximum illumination to minimum occurs within a range of wavelengths not greater than the distance between the sodium-lines. In other words, the grating at a certain angle of incidence will show one of the D lines, and not the other. Experiments with polarised light have proved that these anomalies are only exhibited when the direction of vibration (electric vector) is at right angles to the ruling. The paper gives a detailed account of the appearance of the spectra at different angles of incidence when the grating is in air and when it is immersed in different liquids. It is shown that the phenomena are not due to interference between disturbances coming from widely separated lines, and the author suggests that the matter must be referred to the form of the groove.

A paper by Prof. R. W. WOOD, "*On the Electrical Resonance of Metal Particles for Light Waves*" (Second Communication), was read by the SECRETARY.

In a previous paper the author has shown that granular deposits of the alkali metals exhibit brilliant colours by transmitted light. These colours were referred provisionally to the electrical resonance of the minute particles for light waves. The present paper gives an account of experiments made with gold and silver films to determine whether the resonance is molecular, or whether it is an electrical vibration of metallic masses, smaller than the light waves, though of the same order of magnitude. Further investigations on the dispersion of the films and a more careful study with polarised light will doubtless throw light on the matter.

Prof. H. L. CALLENDAR showed a Simple Apparatus for Measuring the Mechanical Equivalent of Heat.

A cylindrical brass calorimeter is rotated by hand about a horizontal axis at a moderate speed. Unequal weights are suspended from the ends of a belt slung over the cylinder. Stability of equilibrium is secured by making the two halves of the belt of different materials and connecting the side having the smaller coefficient of friction to the larger weight. The weights are adjusted by trial to suit the friction of the belt, and may be varied within wide limits, provided that the ratio is kept nearly the same. The advantages of the methods are:—1. The friction is very nearly independent of the speed. 2. The balance is automatic; the belt immediately adjusts itself to any change of load or friction. 3. There is no change in the thermal capacity of the calorimeter with change of speed or load. 4. There is no pull or bearing-friction to introduce errors. 5. There are no forced vibrations, and no dashpot is required. 6. The actual torque may be measured statically if desired. The rise of temperature is observed by means of a bent platinum thermometer inserted through a hollow axle on one side. The external loss of heat can be eliminated by Rumford's compensation method, or by performing two experiments with different loads on the belt. The motion of the surface of the calorimeter eliminates

the effect of draughts and convection currents, so that the heat loss is much more regular than if the surface were at rest.

The Society then adjourned until October 24th, 1902.

NOTICES OF BOOKS.

Sewage Works Analysis. By GILBERT J. FOWLER, M.Sc. (Vit.), F.I.C., Superintendent and Chemist, Manchester Corporation Sewage Works. London: P. S. King and Son. New York: John Wiley and Sons. 1902. Pp. 135.

PROBABLY at no class of works is analysis of more importance than at those erected for the treatment and disposal of sewage. The work, moreover, like that at water-works, must be continuous, as any cessation in the necessary constant care and attention, without doubt would be followed quickly by pollution of the river or estuary into which the effluent is discharged.

The volume now before us gives an account of the methods of analysis in use in the laboratory of the Manchester Corporation Sewage Works; and, through the courtesy of Mr. Scudder, the author has also been able to include descriptions of some of the more important processes employed in the laboratory of the Mersey and Irwell Joint Committee.

In general, it may be stated that the Manchester methods are for the analysis of a large number of samples of sewage and effluents of the same general character, while the Joint Committee's methods are designed for cases where samples from different works have to be critically examined.

The book is divided into eight chapters and four tables. The first chapter deals generally with the chemical control of sewage purification processes. These processes may be broadly divided into two classes:—

- (a) Mechanical or disposal processes.
- (b) Biological or purification processes.

The first head includes such methods as the removal of garbage and detritus by catch-pits and screens, and the settling out of suspended matters in sedimentation tanks; certain chemicals are used, such as lime, sulphate of alumina, or lime and copperas, and eventually a clear effluent is left.

The biological processes are more modern than the former class just referred to. These processes make use of the micro-organisms originally present, or capable of development in the sewage, to entirely change the organic impurities in the sewage and convert them into harmless inorganic substances.

The remaining chapters deal with the estimation of the various constituents of sewage matter, and the elements generally present therein, such as ammonia, absorbed and dissolved oxygen, nitrates and nitrites, chlorine, acidity and alkalinity, iron compounds, &c., and the analysis of gases from the septic tank and from bacterial filters. The two latter analyses afford valuable information as to the progress of the respective processes.

Finally, there are a few pages of so-called useful tables of atomic weights, weights and measures on the metric system, some conversion tables, and a fairly good index. The book is well and clearly written, and will be useful in laboratories where this class of work is undertaken; it is also adorned with a number of full-page plates illustrating some of the special instruments employed in the work.

Great Britain, Her Finance and Commerce. Morning Post Souvenir Edition. London: The Morning Post. 1901. Pp. 606.

OWING to the failure of a certain person to fulfil his engagements with regard to a large number of advertise-

ments, obtained by the unauthorised use of the name of the *Morning Post*, the proprietor of that well-known paper determined to publish the projected volume at his own cost, thus giving those firms the advertisement they had paid for.

The volume is published for gratuitous distribution through the British Consuls, Exchanges, principal hotels, steamship companies, &c., in all parts of the world, and from a close examination of the book we are bound to admit that it is of far greater interest than most gratuitous literature we have come across at similar places.

There, are, of course, many advertisements, but at the same time there is much more space devoted to interesting and well illustrated articles on a variety of subjects; there are, in fact, forty such articles, each written by an expert. Among these we may call attention to "British Shipping," by Major Jones; "Electrical Industries," by Conrad Cooke; "By-Products from Coal," by Samuel Rideal; "Twentieth Century Sanitation," by J. Priestley; besides others on such diverse subjects as Cold Storage, Construction Material, Hats, Agriculture, Cycles, Locomotives, Chemical Manufactures, and Linen.

There are also a number of full-page plates illustrating the Houses of Parliament, the *Morning Post* Offices, and the town halls and principal buildings in most of our large cities.

The book is well printed on good paper and handsomely bound.

The Tokyo Imperial University Calendar, 2561-62 (1901-1902). Tokyo: Published by the University. 1902. Pp. 328.

NOTHING shows better evidence of the enormous advance made by our "New Allies" during the past half century than the calendar now published yearly by the Tokyo University. In appearance it is almost the counterpart of the calendars issued by our own colleges, and apparently comprises all the subjects taught at home. With the exception of the final page the whole is printed in English and in English characters, and does great credit to those responsible for its production.

Practical Methods of Urine Analysis, for Chemists and Druggists, with Notes on the Composition of the Normal and Abnormal Renal Secretions. Second and Enlarged Edition. Published at the Office of the *Chemist and Druggist*, London; also Adelaide, Melbourne, Sydney, and New York. 1902. Pp. 88.

THE present edition of this book covers about the same ground as the first, which was published in the year 1899, but it has been re-written to a great extent. It is primarily intended as a guide to chemists and druggists in a branch of work which is becoming more necessary to medical diagnosis; we cannot agree, however, with the editor that "no class of men is better fitted to fill this office than are pharmacists"; the business of a pharmacist is to dispense medicines and not to diagnose cases.

Still the book is a very useful one of its kind, and has been enlarged to the extent of thirty pages; it also includes a short formulary of reagents and processes, as well as much new matter. The index is three times the size of that in the first edition, which fact indicates that more care has been taken in its compilation as well as the extent of the revision the book has undergone.

Text-book of Physiological and Pathological Chemistry. By G. BUNGE. Translated by FLORENCE A. STARLING, and Edited by ERNEST H. STARLING. London: Kegan Paul, Trench, Trübner and Co., Ltd. 1902.

THIS text-book of pathological and physiological chemistry, intended for the use of physicians and students, is a translation, most ably performed, of the latest German edition. The style in which it is written is attractive and

stimulating, and cannot fail to inspire the beginner with interest. References to the original papers are fully given on all important or disputed points of the subject, so that the book may be made the basis of exhaustive studies of an advanced nature; great importance is rightly attached by the author to the student's referring constantly to the original literature, and drawing his own conclusions from it. The subject-matter is divided into twenty-nine lectures, arranged in such a way as to provide a complete course of physiological chemistry, and the editor has pointed out cases where Prof. Bunge's views differ from those of the majority of physiologists.

Naturlehre. ("Nature Study"). By Dr. ALOIS LANNER. Wien: Jos. Roth'sche Verlagsbuchhandlung. 1902.

THIS text-book of elementary natural science covers the German official syllabus of scientific teaching issued in February, 1900, and is adapted for the use of the higher classes in intermediate schools. It deals with the elements of mechanics, physics in all its branches, and chemistry, but it cannot be said that the spaces allotted to the various subjects are very evenly balanced. Some thirty pages only are devoted to the whole of chemistry; in these pages not only a brief review of the theory is given, but also an attempt is made at a detailed treatment of the inorganic elements and compounds, and organic chemistry. As might be expected, the result is the merest sketch of the subject. On the other hand, the theory of light is treated far more fully, the pages devoted to polarisation containing a specially good account of that phenomenon. Under Elementary Mechanics is included all that is usually found in a school text-book; only the minimum amount of mathematical knowledge is required in this section. In electricity and magnetism great stress is laid upon practical applications; in fact, this may be said to be a feature common to every section. The book concludes with brief outlines of astronomy and physical geography, but botany and zoology are omitted.

First Book of Qualitative Chemistry. By ALBERT B. PRESCOTT, Ph.D., and EUGENE C. SULLIVAN, Ph.D. New York: D. Van Nostrand Company. 1902.

THIS is the eleventh edition of a book first published in 1879, and has been entirely re-written by the authors. The object of the book is to familiarise the beginner with the present theories of water solution and the law of mass action, and at the same time provide a first course in qualitative analysis. Thus the student who works through the book will not only be conversant with the methods of separation of the most commonly occurring elements and be able to analyse mixtures of inorganic salts, but he will also be acquainted with the main features of the ionic theory, and modern notions concerning solubility and hydrolysis. The introductory sections vary extremely in difficulty. Thus, the rudimentary principles of chemical nomenclature and notation are fully explained, the student being presumably supposed to be ignorant of the most simple formulæ, while some of the sections are really stiff reading, and will very probably prove somewhat bewildering to the beginner. On the other hand, the suggested exercises on the metals of the various groups are most valuable; the advantage of always using solutions of known strength (generally 5 N) cannot be too strongly emphasised, and the tests for delicacy of precipitation should be very useful in encouraging accuracy of observation and manipulation.

Alloys of Cadmium and Magnesium.—O. Boudouard.—The alloys of cadmium and magnesium are all of a white colour more or less brilliant. It is difficult to obtain a perfectly polished surface for microscopic examination. They break if subjected to repeated hammering.—*Comptes Rendus*, cxxiv., No. 24.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 24, June 16, 1902.

Polymerisation and Heat of Formation of Zinc Oxide.—M. de Forcrand.—The numbers obtained by different investigators for the heat of formation of zinc oxide vary from 83.28 cal. to 88.20 cal., the variation being about 5 cal. The author re-investigates this matter in order to elucidate the constitution of the hydrates of zinc peroxide, which substances he has recently been examining. He finds, starting from solid zinc and oxygen gas, the heats of formation of the oxides form a series:—+80.29 cal., +82.98 cal., +84.30 cal., and +84.70 cal. It is thus seen that the series is an increasing one, and this can be explained if it is admitted that the oxide polymerises when it is heated, the reaction being exothermic. It is not reversible, and the oxide when heated to a bright red heat and then cooled gives the same number and preserves a stable state of polymerisation, even if cooled slowly and preserved for a long time. Such polymerisation evolves +4.41 cal.

Compounds of Sulphuretted Hydrogen with Anhydrous Aluminium Chloride.—E. Baud.—Wöhler showed that anhydrous aluminium chloride, when sublimed in a current of sulphuretted hydrogen, retains a certain quantity of this gas. The product obtained has never been analysed. The author therefore investigates this substance to find whether the sulphohydric compounds of aluminium chloride are comparable to hydrates or ammoniacal compounds. He finds that under the action of liquid hydrogen sulphide—(1) a compound, $Al_2Cl_6 \cdot H_2S$, stable at ordinary temperatures, is formed; (2) a compound, $Al_2Cl_6 \cdot 2H_2S$, which is dissociable towards -45° , is formed. It is possible that the solution of Al_2Cl_6 in liquid H_2S contains a compound still richer in sulphuretted hydrogen.

Isomerism in the Benzylidene-menthones, and Preparation of an α -Methyl- α' -isopropyl-adipic Acid Identical with Dihydrocamphoric Acid.—C. Martine.—From the oily products which are formed during the different methods of preparation of benzylidene-menthone which the author previously described, he now separates two compounds which apparently have a definite composition, as seen by their crystalline form and by their fusing-point, and which correspond to the composition of benzylidene-menthone. The one crystallises in tables fusing at 51° , is soluble in ether, alcohol, petrol ether, &c. In a 6 per cent solution of ethylic alcohol its rotatory power is $[\alpha]_D = -185.50$; its oxime is found in the form of fine needles fusing at 172° . The other compound, which was only obtained in small quantities, crystallises in long needles fusing at 47° . It is more soluble than the former compound in the same solvents. Its rotatory power under the same conditions is $[\alpha]_D = -258.5^\circ$. Its oxime has the same appearance as that of the body melting at 51° . This melts at 153° .

Pyromucic and Isopyromucic Acids. Action of Phosphorus Perchloride and Phosphoryl Chloride.—G. Chavanne.—The action of phosphorus perchloride on pyromucic acid was investigated by Lièss-Bodart. He obtained a liquid boiling at 170° , forming the acid under the action of water, and giving an amide melting at 141 – 142° under the action of ammonia. The author repeats these experiments and arrives at the same results whether operating without a solvent or in chloroformic or ethereal solution. After complete purification the chloride appears in extremely refracting prisms melting at about 0° . He examines this product with regard to its action on water, alcohol, ammonia, aniline, and hydrazine hydrate. Pyromucic acid behaves as an acid with regard to phosphorus perchloride, the carboxyl group being replaced by COCl. Isopyromucic acid behaves in a totally different manner,

and is apparently not, strictly speaking, an acid like its isomer, but it seems to possess the properties of a body containing the phenolic or enolic function.

MISCELLANEOUS.

Action of Iodide of Ethyl on Nitrate of Silver.—E. Biron.—In the absence of any solvent, iodide of ethyl reacts energetically on nitrate of silver, giving $C_2H_5NO_3$ in almost theoretical amount, and disengaging about 10,000 cal. It is best to pour the C_2H_5I drop by drop on the powdered nitrate of silver mixed with fine sand in a cooled flask. In the presence of solvents the results are different. With alcohol, as has already been shown by Nef, we obtain besides C_2H_5NO , ordinary ether, $(C_2H_5)_2O$, and nitric acid. In the presence of water there are formed $C_2H_5NO_3$, C_2H_5OH , and HNO_3 . Nef explained the production of $(C_2H_5O)_2O$ by the formation of a product of transitory dissociation, $CH_3 \cdot CH$, which unites with the HNO_3 and the C_2H_5OH , giving $NO_3 \cdot C_2H_5$ and $(C_2H_5)_2O$; but this explanation cannot be admitted as it presupposes a unimolecular reaction, while the measurements of the speeds show that the action of the nitrate of silver on C_2H_5I and on C_2H_5Br are bimolecular reactions. The formation of ether, $(C_2H_5)_2O$, and of products of saponification of nitrate of ethyl in much greater quantities than those which result from the saponification of $NO_3 \cdot C_2H_5$ by water, are attributed by the author to the action of alcohol and water on nitrate of ethyl in the nascent state.—*Journ. Soc. Phys. Chim. R.*, vol. xxxii., p. 667.

Oxidation of some Non-saturated Acids by a Mixture of Sulphuric Acid and Persulphate of Ammonium.—A. A. Albitzky.—In a previous paper (*Bull. Soc. Chim.*, vol. xxii., p. 695) the author showed that by the successive action of $HClO$ and KOH on oleic, elaidic, erucic, and brassidic acids he obtained dioxysearic and dioxybenic acids different from those obtained by direct oxidation by means of permanganate of potash in alkaline solution, or by the action of Ag_2O on the dibromides of the non-saturated acids. In the present research, not yet finished, the author has sought for the conditions which influence the production of one or the other of the stereoisomer dioxyacids; he has found that the results are different according as the oxidising medium is acid or alkaline. By using a mixture of concentrated sulphuric acid and $S_2O_8(NH_4)_2$ he obtained:—With oleic acid, dioxysearic acid fusible at 99.5° ; with elaidic acid, a dioxyacid fusible at 136.5° ; with erucic acid, dioxybenic acid fusible at 99 – 100° ; and with brassidic acid, a dioxyacid fusible at 131 – 133° .—*Journ. Soc. Phys. Chim. R.*, vol. xxxii., p. 640.

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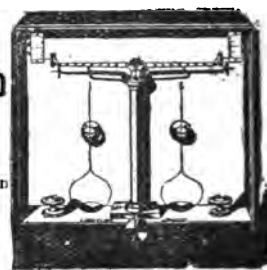


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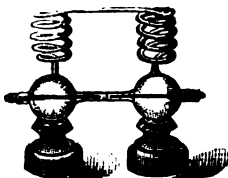
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THE CHEMICAL NEWS.

VOL. LXXXVI., No. 225.

NINTH ANNUAL REPORT OF THE COMMITTEE ON ATOMIC WEIGHTS. DETERMINATIONS PUBLISHED IN 1901.*

By F. W. CLARKE.

THE year 1901 has not been very prolific in researches upon atomic weights, at least so far as actual publications would seem to indicate. A fair amount of work, however, is announced from various sources, and doubtless it will appear in print during 1902. The investigations of current date are summarised in the following pages.

Nitrogen.

Scott (*Journ. Chem. Soc.*, 1901, lxxix., 147) has carefully re-determined the ratios between ammonium bromide and silver. The silver was purified by various methods, and each experiment is cited with the proper details upon this point. The ammonium bromide was prepared from hydrobromic acid, with ammonia from different sources. It was brilliantly white, and remained so upon heating to 180°, whereas that used by Stas in his investigations became greyish. All weights were reduced to a vacuum, and calculations were made with Ag=107.93. Results as follows, but for details the original paper should be consulted:—

Weight NH ₄ Br.	Weight Ag.	Molecular weight NH ₄ Br.
4'89631	5'39380	97'975
2'45925	2'70914	97'972
3'29478	3'62928	97'982
4'46957	4'92273	97'994
4'20661	4'63303	97'996
4'23664	4'66644	97'989
4'31464	4'75175	98'001
6'19233	6'82047	97'990
8'77664	9'66608	97'999
10'47233	11'53416	97'994
4'91997	5'41834	98'0028
5'00442	5'51164	97'997
5'17914	5'70390	98'000
4'84099	5'33177	97'995
5'10677	5'62515	97'984

Rejecting the first three experiments, in which the bromide was slightly acid, Scott's mean value for the molecular weight of NH₄Br is 97.995. Stas found 98.032, and Scott supposes that his salt may have contained traces of platinum, whence the greyiness which appeared on heating.

As a further check on the determinations Scott collected and weighed the silver bromide produced, and so determined anew the ratio between Ag and AgBr. Data as follows:—

Weight Ag.	Weight AgBr.	Ratio AgBr to 100 Ag.
6'82315	11'87733	174'074
9'66809	16'82816	174'059 (a)
5'41906	9'43315	174'0735
5'51258	9'59596	174'074
5'70686	9'93346	174'062
5'33191	9'28093	174'064
5'62572	9'79254	174'067

(a) 174.090 when corrected for a known impurity.

Stas' value for this ratio is 174.080.

Two additional experiments were made upon the ratio between ammonium chloride and silver.

Weight NH ₄ Cl.	Weight Ag.	Molecular weight NH ₄ Cl.
4'78257	9'64484	53'519
5'51744	11'12810	53'513

In still another experiment connected with these two, the silver chloride was weighed. 4.7850 NH₄Cl gave 12.82048 AgCl. Hence NH₄Cl=53.5164. Stas' value is 53.532. Further investigations are promised; but until they are complete Scott regards it as premature to compute the atomic weight of nitrogen from these data. As they stand they give:—

From the bromide		NH ₄ = 18.040
From the chloride		NH ₄ = 18.059

Calcium.

The atomic weight of calcium has been re-determined by Hinrichsen (*Zeit. Phys. Chem.*, xxxix., 311). The purest Iceland spar was ignited, and the ratio so determined between CaCO₃ and CaO. A correction is applied for 0.032 per cent of Fe₂O₃, found by analysis, and assumed to represent FeCO₃ in the original mineral. Weights were reduced to a vacuum, and calculations were made with O=16 and C=12. The data are as follows, all corrections applied:—

Weight CaCO ₃ .	Weight CaO.	Atomic weight Ca.
30'72157	17'22354	40'144
32'77791	18'37587	40'141
34'45625	19'31698	40'142
33'36885	18'70723	40'141
Sum, 131'32458	73'62462	40'142

Herzfeld's determinations of the atomic weight of calcium, cited in the report of last year, have been reproduced in the *Berichte* (vol. xxxiv., p. 559), and so made accessible to the general reader.

Arsenic.

Somewhat elaborate determinations of the atomic weight of arsenic have been made by Ebaugh (Doctoral Thesis, University of Pennsylvania, 1901) under the guidance of Edgar F. Smith. First, silver arsenate was converted into silver chloride by heating in hydrochloric acid gas. All weights were reduced to a vacuum. The results are as follows, with O=16, Cl=35.45, and Ag=107.92.

Weight Ag ₃ AsO ₄ .	Weight AgCl.	Atomic weight As.
0'23182	0'21547	74'987
0'47996	0'44615	74'944
0'52521	0'48820	74'956
0'80173	0'74517	74'996
0'94782	0'88083	75'061
1'02047	0'94830	75'083
1'03558	0'96258	74'974
1'05462	0'98014	75'033

Mean 75'004

The silver chloride from seven of these experiments was next reduced by heating in hydrogen. The silver contained in the arsenate was thus determined, giving the following data:—

Weight Ag ₃ AsO ₄ .	Weight Ag.	Atomic weight As.
0'23182	0'162175	75'027
0'47996	0'33583	74'950
0'52521	0'367525	74'907
0'80173	0'56099	74'936
0'94782	0'66318	74'959
1'02047	0'71400	74'964
1'05462	0'73771	75'082

Mean 74'975

* Journal of the American Chemical Society, vol. xxiv., No. 3.

Experiments upon the conversion of silver arsenate into bromide were unsatisfactory. The conversion of lead arsenate into lead chloride gave good results, however, as follows:—Calculated with Pb=206.92.

Weight $Pb_3As_2O_8$.	Weight $PbCl_2$.	Atomic weight As.
0.38152	0.35381	74.988
0.436197	0.40449	75.016
0.57218	0.53065	74.964
0.60085	0.55717	75.020
0.74123	0.68736	75.010
0.77107	0.71494	75.067
0.88282	0.81858	75.054
0.97779	0.90674	75.054

Mean 75.022

Three more determinations, based upon the conversion of lead arsenate into lead bromide by heating in gaseous hydrobromic acid, were also successful. Calculated with Br=79.95.

Weight $Pb_3As_2O_8$.	Weight $PbBr_2$.	Atomic weight As.
0.59704	0.73092	75.066
0.61712	0.75567	74.967
0.65799	0.80569	74.980

Mean 75.004

The general mean of all twenty-six determinations is—

As = 75.008.

Various other methods of determination were attempted, but unsuccessfully.

Antimony.

Friend and Smith (*Journ. Am. Chem. Soc.*, 1901, xxiii., 502) have applied a new method to the determination of the atomic weight of antimony. Potassium tartrantimonite (tartar emetic) was heated in dry hydrochloric acid gas, the final residue of the operation being potassium chloride. All weights were reduced to a vacuum. The results were as follows, when H=1.008, C=12, K=39.11, and Cl=35.45.

Weight of salt.	Weight KCl.	Atomic weight Sb.
1.19481	0.27539	120.345
1.57004	0.36186	120.359
2.00912	0.46307	120.351
2.04253	0.47073	120.379
2.16646	0.49935	120.341
2.25558	0.51982	120.385
2.61255	0.602155	120.350
2.95272	0.68064	120.311

Mean 120.353

This value approximates to those found by Schneider and by Cooke from their studies of antimony trisulphide.

(To be continued).

SPECIFIC WEIGHTS OF VARIOUS TIMBERS.

By E. G. CLAYTON, F.I.C., F.C.S.

DETERMINATIONS have been made of the relative densities (water = 1) of the following varieties of seasoned timber. The weighed blocks had been accurately sawn and planed to as nearly as possible identical cubic dimensions, namely, 8×3×2 inches.

Of course, the figures do not express the actual specific gravities of the woody substance deprived of water, which would approach 1.2 to 1.5, but "the sum of the weight of the solid matter with that of the water contained in it, and of the air of the pores" (Groves and Thorp, "Chemical Technology," 1889, i., 6).

The specimens had been carefully chosen for a small museum of economic botany, and were thoroughly representative; they are named in the list in the order of

their densities, and roughly classified as soft, hard, or medium woods.

Genus and species of tree.	Ordinary name of tree or wood.	Relative density (water=1)	Nature of wood.
<i>Pinus strobus</i>	Quebec or yellow pine	0.433	Soft
<i>Populus fastigiata</i> ..	Lombardy poplar	0.452	"
<i>Abies excelsa</i> (Norway or European spruce fir), varie-	Yellow deal (1) ..	0.452	"
ties of	Yellow deal (2) ..	0.471	"
	White deal ..	0.471	"
	Yellow deal (3) ..	0.490	"
<i>Abies nigra</i>	American spruce fir	0.509	"
<i>Abies Douglasii</i> ..	Oregon or Douglas pine	0.529	"
<i>Tilia Europæa</i> ..	Lime or linden ..	0.548	"
<i>Populus alba</i>	Abele or white poplar	0.558	"
<i>Juglans regia</i>	Walnut	0.625	Medium
<i>Ulmus campestris</i> ..	Elm	0.625	Hard
<i>Larix Europæa</i> ..	Larch	0.625	Medium
<i>Quercus robur</i> (Pedunculata)	British oak ..	0.635	Hard
<i>Pinus Australis</i> ..	Southern or pitch pine	0.654	Soft
<i>Quercus robur</i> (Sessiliflora)	Austrian oak ..	0.663	Hard
<i>Prunus cerasus</i> ..	Cherry	0.673	Medium
<i>Betula alba</i>	Birch	0.673	"
<i>Quercus cerris</i> ..	Turkey oak ..	0.673	Hard
<i>Fraxinus excelsior</i> ..	Ash	0.683	Medium
<i>Swietenia mahagoni</i>	Honduras mahogany	0.683	Hard
<i>Robinia pseud-acacia</i>	Acacia	0.692	Medium
<i>Pinus sylvestris</i> ..	Scotch fir	0.702	Soft
<i>Pinus sylvestris</i> ..	Memel fir	0.711	"
<i>Fagus sylvatica</i> ..	Beech	0.740	Hard
<i>Neandandra rodiei</i> ..	Greenheart ..	0.759	"

Chemical Laboratory,
32, Holborn Viaduct, E.C., July 8, 1902.

COMPOUNDS OF CHLORIDE OF BISMUTH WITH THE ORGANIC BASES.

By L. VANINO and O. HAUSER.

CHLORIDE of bismuth is dissolved without change in acetone, and the solution treated with organic bases gives precipitates. In the case of aniline and other aminobenzenic bases we obtain amorphous deposits, slightly nitrated and badly defined, but with other bases we obtain well characterised crystalline precipitates which may be, according to the conditions, compounds of $BiCl_3$ either with the hydrochlorate of the base or with the free base.

Chloride of Bismuth and Quinolein, $BiCl_3 \cdot C_9H_7N$.—An excess of the base poured into the acetonic solution of $BiCl_3$ gives a thick crystalline mass, slightly soluble in acetone. The salt is hardly changed by water; if in the preparation the chloride of barium contains a little free acid the salt is contaminated with the double chloride, $BiCl_3 \cdot 2(C_9H_7N \cdot HCl)$, which is also slightly soluble.

Iodide of Bismuth and Quinolein, $BiI_3 \cdot C_9H_7N$.—The preceding salt added to a boiling aqueous solution of iodide of potassium gives a deep red liquor, which, after cooling, deposits a red precipitate of the corresponding iodide.

Chloride of Bismuth and Hydrochlorate of Quinolein, $BiCl_3 \cdot C_9H_7N \cdot HCl$.—The salt $BiCl_3 \cdot C_9H_7N$ dissolves easily in dilute hydrochloric acid, and on evaporation the solution gives crystals of the double chloride.

Chloride of Bismuth and Pyridine, $2BiCl_3 \cdot 3C_5H_5N$.—This has been already described by Montemartini (*Gazz. Chim.*, 1900, p. 493), but is obtained as easily in the form

of a white powder by the author's method; it is non-hygroscopic, slightly changed by water, and decomposed by heat into chloride of bismuth and pyridine.

Iodide of Bismuth and Pyridine, $\text{BiI}_3 \cdot \text{C}_5\text{H}_5\text{N}$.—The same method of preparation is used as for quinolein. It occurs as a deep red powder, finely crystalline; it is slightly soluble in warm alcohol or in a solution of iodide of potassium; it is deposited from this latter solution, on cooling, in small needles.

Chloride of Bismuth and Hydrochlorate of Pyridine, $2\text{BiCl}_3 \cdot 3(\text{C}_5\text{H}_5\text{N} \cdot \text{HCl})$.—The compound of chloride of bismuth and pyridine dissolves in dilute hydrochloric acid, and the solution gives, on evaporation, beautiful needles of the double salt; this salt is decomposed by water, giving a deposit of BiOCl .

Chloride of Bismuth and α -Naphthylamine, $3\text{BiCl}_3 \cdot 2\text{C}_{10}\text{H}_7\text{NH}_2$.—A concentrated solution of α -naphthylamine in acetone, poured in excess into an acetonic solution of chloride of bismuth, furnishes a white crystalline precipitate, slightly soluble in dilute hydrochloric acid and in acetone; it is decomposed by water, and also by an excess of hydrochloric acid; in this latter case BiCl_3 and $\text{C}_{10}\text{H}_7\text{NH}_2 \cdot \text{HCl}$ are formed.

Iodide of Bismuth and α -Naphthylamine, $\text{BiI}_3 \cdot 2\text{C}_{10}\text{H}_7\text{NH}_2$.—Proceed in the same manner as with pyridine and quinolein; the blood-red liquor is filtered hot to separate a deep brown resin, then evaporated in the desiccator, where it gives long deep red needles arranged in bunches; this body cannot be washed without decomposition, and, further, the return is very small.—*Berichte*, vol. xxxiv., p. 416.

RECENT DEVELOPMENTS IN COLOURING-MATTERS.*

By Geheimrath Professor OTTO N. WITT, Ph.D. F.C.S.,
of Berlin.

(Concluded from p. 16).

SOME of the older synthetical methods of producing indigo are so easy and rapid that they can easily be shown as a lecture experiment. If, for instance, we add a caustic potash solution to a solution of orthonitrobenzoic aldehyde in acetone, indigo is formed at once and settles out in dark blue crystalline flakes (Exp. IX.). The synthesis now in practical use is a little more delicate in its execution, but there are certain modifications of it which are rapid enough to be shown in a lecture experiment (Exp. X.).

The action of the alkali on the phenylglycin-carbonic acid does not at once produce indigo, a colourless derivative of the dye, indoxylcarbonic acid, or rather its potash salt, is formed at first; but if we dissolve this in hot water, and introduce a current of air, it is at once, and with a quantitative yield, transformed into indigo which settles out in the shape of a crystalline deposit of infinitely fine division. This is collected in filter-presses, and delivered into commerce in the shape of a paste or a powder.

The industrial synthesis of indigo is extremely interesting, because it is a triumph not wholly due to chemical science. Science has shown the way to success, but it was quite unable to clear away the difficulties arising from practical and economic considerations. Here the representatives of our great industry had to advance independently and on paths for which theoretical knowledge could not serve them as a guide. Unlimited praise and admiration is certainly due to them for the masterly way in which they grappled with colossal difficulties, and for the courage with which they staked millions on the realisation of one great idea.

At the same time we cannot help feeling some regret for the indigo planters in the far East, who, after enjoying

more than a century of easy prosperity, see now that more serious times are in store for them. They see the day coming when the indigo plantations will disappear, in the same way in which the madder fields of Avignon have vanished. But we are consoled by the knowledge that, especially for India, the time has already come, which has been so vividly described by Sir William Crookes, in one of his addresses to the British Association, as the future in store, sooner or later, for all humanity, the time when bread begins to be scarce. It seems to me that any one who, by bringing about some great commercial revolution such as we have seen in the indigo trade, causes land in India to become free for the growing of rice and other cereals, renders a great service to large numbers of poor natives, and need therefore not be blamed for lessening to some extent the prosperity of a class of people who have had an unusually good opportunity of accumulating wealth in the past.

It is a strange fact, that with indigo the history of the fight of the madder root against artificial alizarine is almost literally repeated in spite of the great difference of original conditions in the two cases. Madder was a product containing at its best only 4 per cent of actual colouring matter; the rest was useless fibre and obnoxious impurities which greatly hampered the dyer in his work. Alizarine, entering into competition with this natural product, was, on the contrary, the colouring matter in a pure state, and therefore not only cheaper but also much easier in its application. Indigo, such as we receive it from India and Java, is a manufactured article, the best qualities of which contain 50, 60, or even 70 per cent of pure dye-stuffs, besides impurities which have always been considered as perfectly harmless. Thus the artificial product did not seem to have much scope for improvement as far as the quality came into consideration. Here again we have committed a mistake. We know now that the impurities are not harmless, and that the blues dyed with artificial indigo are quite as superior in brightness and purity of shade to those obtained with natural indigo, as alizarine reds were to madder reds. This has, however, not always proved to be an advantage for the manufacturers of artificial indigo. The world does not ask for bright indigo shades, and a good many prejudices in that respect had to be overcome before artificial indigo was admitted as a legitimate substitute for the natural product in some of its most important applications. Yet a simple consideration will show that it is always easy to deteriorate the brilliancy of a dyed shade, whereas no art of the dyer will suffice to produce brilliant shades on textile fabrics with dye-stuffs that carry their share of dirty admixtures within them.

In its application to the fibre, indigo is perhaps the most remarkable of all dye-stuffs, for it is the principal representative of that extraordinary class of colouring-matters which must be applied by the vat process. This process, which consists in first reducing the dye-stuff into a leuco-compound before applying it to the fibre, on which the original colouring-matter is formed again by the action of the oxygen of the air, seems to have nothing in common with the ordinary dyeing processes. If, however, we consider it more closely, we come to the conclusion that vat colours are a class of dye-stuffs in which the functions of dyeing and of selective absorption of light are distributed on two different forms of the substance, one of which contains two atoms of hydrogen more in its molecule than the other.

This theory is supported to some extent by the fact that, what we are pleased to call leuco-compounds are in the majority of cases by no means colourless. Indigo-white itself is not white but yellow in its alkaline solution which we call a vat. Other vat-dyes have leuco-compounds which are even more strongly coloured. Thus the leuco-compound of indanthrene, a beautiful new colouring-matter, is blue like indanthrene itself; flavanthrene, a yellow dye-stuff, which has not yet left the laboratory of its inventor, has a blue leuco-compound. One may say,

* A Lecture delivered at the Royal Institution of Great Britain, Friday, March 21, 1902.

that with all vat-colours the real dye-stuff is the leuco-compound which is afterwards, when once fixed on the fibre, transformed into a pigment by the oxidising influence of the air.

In 1825 Faraday discovered, in this very house, benzene; the original specimen, prepared by his own hands, is before you. We look upon it reverently, like on a sacred relic bequeathed to us by a master-mind. But what a development has sprung from this first attempt to unravel the mysteries of the aromatic series! Our science as well as our industry have been revolutionised by the investigation of the derivatives of benzene, and the world has been embellished by the gay and brilliant dyes of which it is the mother-substance. The study of the chemistry of these dye-stuffs has become a domain of science which, for variety and fascination, can hardly be surpassed by any other. The deeper we penetrate into it the more it proves an inexhaustible mine of the most subtle scientific thought, yet one which never loses touch with practical life; it is interesting alike to the philosophical mind that wishes to revel in the wonderful perfection and order of nature, and to the philanthropic spirit which rejoices in seeing many thousands of hands occupied and princely fortunes produced by the utilisation of what was only a short time ago a refuse and an encumbrance. It teaches a lesson even to those who are not attached to science and apt to consider it as a kind of pastime for people who lack ability for practical life. For they cannot help seeing that, in this case, the most intricate science has led to something eminently practical, commensurate to a standard which, though unknown to the Bureau International des Poids et Mesures, is to some people the only reliable one, viz., the one of £ s. d.

I am afraid that the high praise which I feel justified in bestowing on what has been the favourite pursuit of my life is not fully substantiated by the contents of this lecture. The subject which I had to treat is so vast, that all I have been able to say is nothing but a sketch or a programme of what would require a long series of lectures if full justice were to be done to it. My one excuse for attempting to sketch, in the short space of one hour, so vast a subject, is the place in which I had the honour to speak. An audience that has been addressed more than once by the pioneers of the chemistry of dye-stuffs, by Faraday, Hofmann, William Perkin, and others, could, from one of the Epigones, not have looked for more than a few notes and additions.

NOTES ON IRON ANALYSIS.

By GEORGE T. DOUGHERTY, Chicago.

Complete Evolution Method for Sulphur in Iron.

It has been a matter of knowledge among many of us since about ten or fifteen years ago, though it has never been promulgated in text-books issued to date, that the evolution method frequently fails to give the full amount of sulphur present in samples of pig- or cast-iron, the deficiency ranging from 0.005 up to 0.025 per cent, which is the maximum that I have obtained in my individual experience. Various unsuccessful attempts have been made to overcome this defect of the method, in order to avoid resorting to the slow and tedious gravimetric oxidation method. Among them has been the use of hot dilute hydrochloric acid instead of cold for starting the decomposition, the treatment of the insoluble residue after evolution for any additional sulphur, and the addition of an arbitrary correction factor of 0.010 to 0.020 to the per cent obtained by direct titration. This is the custom in many places, a practice which involves a serious element of uncertainty when it comes to the question of accepting or rejecting a car lot of pig-metal bought on the specification of not to exceed 0.030 or 0.050 per cent of sulphur,

as the case may be. The insoluble residue rarely yields on examination enough sulphur to account for the shortage. Most of the lost sulphur is supposable of the organic or mercaptan series, which is not absorbable in the alkali solution.

Recently there came to my notice a method proposed by Walters and Miller of annealing iron drillings in a porcelain boat in a porcelain or nickel tube in a current of natural gas or some non-oxidising gas, previous to solution in the evolution flask. The principle of the method, together with the closely agreeing analyses submitted by the authors in corroboration, appeared attractive to me, but the mode of application required would put it out of the way of many crowded laboratories which may not care to instal such a bulky apparatus. My mind chanced to hit on trying a covered porcelain crucible in its stead, and I have found it to work like a charm, no current of any kind of gas being necessary, but only a piece of filter-paper loosely laid on top of the drillings in order to maintain a reducing atmosphere during the operation of igniting for fifteen minutes. I first tried wrapping the drillings in filter-paper, and found it would require at least one-half hour's ignition to get out the maximum results. If, however, the drillings are placed direct in the porcelain crucible and one-half of a 9 c.c. filter-paper is laid loosely on top of the mass, fifteen minutes' strong ignition is sufficient. The "adjustable" type of Bunsen burner sold for gasoline gas will give the requisite strong temperature with coal-gas. Heavily graphitic drillings barely need the presence of paper to prevent oxidation, but it is a safe policy to use it on all kinds of iron samples. After the ignition the drillings will be found to have slightly set or caked together, but are easily disintegrated and transferred without loss into the evolution flask. I use 50 c.c. of 1 to 1 hydrochloric acid on 5 grms. of drillings and absorb the H_2S evolved in 17 c.c. of caustic potash solution (350 grms. of Merck's purified stick potash in 2 litres) in a Will and Varrentrapp nitrogen bulb, which is admirably adapted to prevent back suction as well as to insure a double bubbling of the gas. This amount of potash solution, after diluting to the usual volume of 400 c.c. in a No. 4 Griffin beaker, and acidifying with 20 c.c. strong hydrochloric acid, usually takes up 0.1 to 0.35 c.c. of standard iodine solution in a blank test, and of course this must always be deducted. Standardise the iodine solution with a standard steel, whose sulphur contents have been determined by two or more gravimetric oxidation analyses, preferably of over 0.050 per cent sulphur. The iodine solution is made up by dissolving 17.715 grms. of pure iodine in a 50 c.c. solution of 30 grms. of potassium iodide in a beaker, and after all is dissolved by shaking, dilute to 5 litres; each c.c. of it will be found to equal 0.0005 gm. sulphur or 0.010 per cent sulphur when calculated on 5 grms. of steel run through as above. Verify each fresh iodine solution, or whenever in doubt, by running the standard steel through under similar conditions as for regular work. In gravimetric oxidation work on standard or special samples, the addition of 20 c.c. bromine water to 60 or 80 c.c. of the usual aqua regia will effect the complete oxidation of all the sulphur present, which is not usually possible with the simple aqua regia; only one evaporation is necessary.

Below I give a memorandum of my various experiments in this complete evolution method. The three standard cast-irons, B, C, and D, were furnished by the Standardising Bureau of American Foundrymen's Association, with its official analyses of same. The two Lebanon pigs are coppery, 0.8 to 1.25 per cent copper; the gravimetric sulphur percentages given of each were determined by myself.

Standard Iron B.

Official analysis	..	$\left\{ \begin{array}{l} 0.038 \text{ per cent sulphur by evolution method.} \\ 0.056 \text{ per cent sulphur by oxidation method.} \end{array} \right.$
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	Evolution method.
Per cent.	
Not ignited previously	0.041
Wrapped in filter-paper and ignited thirty minutes	0.059
Wrapped in filter-paper and ignited fifteen minutes	0.053
Ignited fifteen minutes without any paper at all	0.049
Ignited fifteen minutes, paper on top of drillings	0.052
	0.055
	0.057
	0.058
	0.057

Standard Iron C.

Official analysis ..	{ 0.059 per cent sulphur by evolution method.
	{ 0.076 per cent sulphur by oxidation method.

	Evolution method.
Per cent.	
Not ignited previously	0.060
Wrapped in filter-paper and ignited thirty minutes	0.076
Ignited fifteen minutes, paper on top of drillings	0.076
	0.079
	0.076

Standard Iron D.

Official analysis ..	{ 0.024 per cent by evolution method.
	{ 0.031 per cent by oxidation method.

	Evolution method.
Per cent.	
Not ignited previously	0.022
Ignited fifteen minutes, paper above drillings	0.031
	0.030

Lebanon Pig, 1.

Oxidation method	0.046
Evolution without previous ignition	0.020
Evolution after one-half hour's ignition	0.043

Lebanon Pig, 2.

Oxidation method	0.032
Evolution without previous ignition	0.010
Evolution after one-half hour's ignition	0.030

Stewart Pig.

Oxidation method	0.011
Evolution without previous ignition	0.003
Evolution after fifteen minutes' ignition	0.009

There always appears an uneven mass of "scum" on the surface of the liquid when iron drillings not previously ignited have been dissolved in the evolution flask. But when the drillings treated had been ignited before the solution would have a smooth surface and be as free of scum as that of steels. In the case of the three cast-iron standards, B, C, and D, the solution after ignition of the drillings takes place very quickly and almost instantaneously; but the Lebanon pigs after ignition require a rather longer time for complete solution than when not previously ignited. I use 20 c.c. water + 35 c.c. strong HCl on Lebanon pigs on account of their peculiar or coppery nature, when ordinary irons require only 25 c.c. water + 25 c.c. strong HCl.

Determination of Graphite by Direct Weight.

One grm. of drillings is treated with 60 c.c. of nitric acid, 1.13 specific gravity, in a No. 2 Griffin beaker; gentle heat is applied for about thirty minutes, or until no more nitrous fumes come off; then boil hard for about five minutes. Weigh previously a Gooch crucible and a disk of Swedish No. 0 filtering-paper cut to about the size of the bottom of the Gooch crucible and just previously dried for one hour at 115 degrees C., weighing the disk after three or five minutes in the desiccator, for it is sometimes

aggravatingly hygroscopic. Stir the liquid just before filtering it into the paper disk fitted in the Gooch crucible under suction. Wash in this order:—Once with diluted nitric acid, thrice with hot water, twice with hot 10 to 15 per cent KHO solution, thrice with hot water, twice with hot dilute hydrochloric acid, thrice with hot water, once with alcohol, and once with ether or 87 degrees test gasoline.

Dry the Gooch crucible and contents for one hour at 115 to 120 degrees C., and weigh after five minutes in the desiccator (1), open the crucible and ignite until nothing black remains, and weigh again (2).

1. Increase of weight over combined tares of Gooch crucible and paper disk equals graphitic matter plus silica, &c.

2. Increase of weight over tare of crucible equals silica or mineral matter.

1—2 equals graphitic matter. Graphitic matter multiplied by 0.96 equals amount of true graphite.

The object of stirring the liquid just before filtering is to prevent occasional clogging of the filter by the gelatinised silica. This, together with the washing with KHO, removes all the necessity for introducing any hydrofluoric acid, as is used by several chemists. Hydrofluoric acid does not decompose or remove any carbide insoluble in simple dilute nitric acid, as KHO does. Hydrofluoric acid, whenever used, usually causes irregular final results. It will be necessary to wash the graphitic residue with KHO, not only to remove silica, but also to decompose the small quantity of insoluble carbide, which will be shown by the deep brown liquid running out of the filter on application of KHO. When the silica is all but eliminated in this manner graphite will be vastly easier and quicker to burn up. I do not recommend asbestos for filtering and burning graphite on for direct weighing, for two reasons:—

1. The burning of graphite on an asbestos filter, since it is out of contact with the red-hot metal of the crucible, is a very difficult and tedious operation in any crucible.

2. Asbestos, even when previously purified by acids and ignited, is, contrary to general expectations, slightly but appreciably volatile at the temperature of the blast-lamp or "adjustable" Bunsen, which temperature is necessary to burn up graphite. I have determined this fact by many experiments on different lots of asbestos.

The use of a Gooch crucible with a paper disk for filtering and weighing graphite and washing, as above recommended, and applying the factor for 0.96 are points of the method which will largely remedy objections heretofore urged against the method of direct weight for graphite. This method professes to give commercially accurate results, though not quite equal in scientific accuracy to the regular combustion process most carefully performed.

Continual Diminution of Graphite in Much Used Drillings.

Dr. P. W. Shimer, in *Transactions of American Institute of Mining Engineers*, vol. xlv., recommends that for determining graphite in cast-iron the drillings be made wet with alcohol to mix and weigh out for analysis to prevent segregations and irregularities in results. I have since found out a startling fact—that if alcohol be not used in mixing the drillings the per cent of graphite (and total carbon also) will be constantly lowered little by little whenever the drillings are poured out of the container, mixed over and weighed out for any determination of that or other elements. Also, a serious loss in graphite will be suffered if drillings of cast- or pig-iron are powdered and sifted, as is done by many chemists, though with the laudable intention of securing a thoroughly homogeneous sample.

To illustrate the extent of loss in graphite contents in dry samples of iron drillings I submit here below the official analyses of standardised iron drillings A and D supplied by the Standardising Bureau of American Foundrymen's Association when freshly prepared, and in

own determinations (by combustion) after the contents of the bottles containing those two samples have been three-fourths or four-fifths used up:—

		Graphite when new. Per cent.	Graphite after three-fourths has been used up. Per cent.
A		3'11	2'12
D		2'80	2'16

It is up to the Standardising Bureau of the American Foundrymen's Association to supply its carbon standards only after mixing with alcohol, and fill the bottles with alcohol-moist drillings.

The probable reason for the constant loss of graphite in dry samples when much used is the low gravity of the graphite as compared with the rest of the iron, and its tendency to separate as fine dust and go off in the air whenever disturbed.

Recovery of Old Copper Solutions for Carbon Determinations.

Copper potassium chloride itself is not more expensive than other C. P. reagents usually used, by the pound, but as so much more of it is required per determination than other reagents are required for the estimation of other elements, it will form quite an item of expense when very many carbon determinations are made through that reagent. One pound of it will be used up in making a comparatively few carbon determinations, about 40 to 50 grms. being required for each determination. It will therefore be found economical to retreat or "revivify" old copper solutions with a view to re-using them for other carbon determinations.

The following plan has been adopted by me after various experiments with excellent results:—To 7 grms. of potassium chlorate already in a 16-ounce flask add 400 of the old copper potassium chloride solution and 40 c.c. strong hydrochloric acid. Heat to boiling, and boil strongly for five or ten minutes. When cool filter through asbestos and it will be ready for re-use. A slight odour of free chlorine will not be hurtful. One-half of the 40 c.c. strong hydrochloric acid used above is necessary to re-dissolve and keep in solution the re-oxidised iron salts, and the other half is calculated to supply the usual 5 per cent of free acid to the solution. After revivifying five or six times it will be advantageous to throw them away, they having then become so nearly saturated with salts as to cause slow filtering on some steels.

Manganese in Iron and Steel.

Standard potassium permanganate solution, 3'90 grms. of the salt in 2 litres of water. Each c.c. equals 0'0010 gm. manganese, or 0'20 per cent of manganese when 1 gm. of sample is weighed out and treated, and one-half of the resultant solution is titrated.

The method:—Treat 1 gm. of drillings with 20 c.c. nitric acid of 1'13 specific gravity for cast-iron, or 15 c.c. nitric acid of 1'20 specific gravity for steel in a No. 2 Griffin beaker. Heat and boil until very low down in the beaker and the bulging centre of the bottom of the beaker almost shows up. Cool and dilute a little with water; transfer with rinsings into a 300 or 400 c.c. volumetric flask. Add emulsion of zinc oxide from a bottle, part by part, shaking the flask all the time, until the iron precipitates stiffly, then add a little more zinc oxide and shake vigorously, the precipitate showing a slight whitish tinge; and fill up with water to the mark. Pour all into a No. 3 Griffin beaker, and stir well with a glass rod. Let settle for a few minutes, and pour the rather (not excessively) milky supernatant liquid carefully into a 150 or 200 c.c. volumetric flask (according as it has been from a 300 or 400 c.c. volumetric flask) up to the delivery mark. One-half of either 300 or 400 c.c. will represent $\frac{1}{2}$ gm. of sample. Pour it from the 150 to 200 c.c. flask into a 16-ounce ordinary flask; bring it to a boil, and titrate as usual with standard permanganate solution. If the burette reads 4'2

c.c. then $4'2 \times 0'2$ equals 0'84 per cent Mn. I have a standard steel of 0'75 per cent manganese which has been determined gravimetrically, with which I check up the standard permanganate solution when freshly made up or whenever in doubt of it, or to determine any correction needed in the routine work. For example, the permanganate solution in use at present would show on test with the standard steel 0'77 per cent manganese instead of 0'75; therefore, I deduct 0'02 from all the results obtained by titration with this permanganate solution.

The advantages of this modified Volhard method are:—

1. No evaporation with sulphuric acid is necessary.
2. This obviates any necessity for filtering off the iron.
3. It is desirable to have a slight excess of zinc oxide present when ready for titrating, as indicated by the moderately milky appearance of the liquid, as it increases the sharpness and permanency of the end reaction in titrating.

In case one is troubled with over-titrations from having to deal with samples running unusually high in manganese (over 2 per cent), and widely differing in high manganese contents not approximately known, it would be practicable to titrate back, as is done in the Gay-Lussac method of assaying silver bullion, with a standard solution of manganous chloride, which may be prepared by evaporating the proportion of 15 c.c. standard permanganate solution down to 3 or 4 c.c., adding a few drops of hydrochloric acid, and boiling as long as chlorine comes off; then neutralising with zinc oxide, and diluting to 10 c.c., when 1 c.c. equals 1 c.c. permanganate solution. This method if preparing a standard manganous chloride solution is due to Dubois and Mixer. If these gentlemen were correct in giving proportions of permanganate solution to convert into a standard manganous chloride solution, I would simplify and save time and labour of evaporating down the very dilute standard permanganate solution by taking of the dry permanganate salt equivalent to 1500 c.c. of standard permanganate solution (2000 : 1500 :: 3'90 : x = 2'975 grms.), dissolving it in 50 c.c. water in a No. 3 Griffin beaker, treating as directed, and finally diluting to 1000 c.c., when 1 c.c. equals 1 c.c. permanganate.—*The Iron Age*, May 8, 1902.

EXPERIMENTAL RESEARCHES ON THE CONSTITUTION OF CRYSTALS.*

By A. E. TUTTON, B.Sc., F.R.S., F.C.S.

EXPERIMENTAL work in connection with the study of crystals offers attractions of a more than usually fascinating kind. For, in the first place, crystals themselves are such wonderfully beautiful objects; they are, indeed, unquestionably the most beautiful of all the inanimate productions of Nature. It is even doubtful whether we are right in considering them inanimate, for the force of crystallisation, though it may lie dormant for thousands of years, is ever ready, when a suitable environment offers, to re-assert itself. In the second place, the study of crystals involves the investigation of that most exquisite of all the forms of energy, light—that extraordinary effect of wave-motion in the ethereal all-pervading medium, which we now believe to be due to an exceedingly rapid, periodic, oscillating change in the electrical condition of the atoms and molecules of the incandescent light-giving source.

We will allow the beam of light from an electric lantern to fall on this cluster of diamonds, carbon in its most exquisite crystalline form. The diamonds are arranged, as you see, in the shape of a crown, and were generously lent for this lecture by Mr. Streeter. You observe a magnificent play of light waves rippling towards you in every variety of colour and scintillation. The object of the experiment is that you may distinguish the two distinct

* A Lecture delivered at the Royal Institution of Great Britain, Friday, May 2, 1902.

types of emanation, namely, exterior reflections of white light from the facets and coloured rays due to penetration of the light into the interior structure of the diamonds, and subsequent refraction and reflection outwards again. The former kind you observe best in the innumerable images in white light of the carbon points of the lanterns, reflected on the ceiling and screen.

It is by the determination of the direction of the exterior reflections that we are enabled to ascertain the wonderfully regular angular relations of the various faces of the crystals; and it is by the study of the light which has penetrated that we gain the best information concerning the internal structure.

We shall consider this evening some of the results of a study of the crystals of certain series of definitely related chemical salts. There has been a great want of correlated work of this kind, having for its definite object the elucidation of the relationship between the chemical composition of a crystallised substance and the peculiar type of symmetry which it exhibits.

The salts which have, up to the present, been studied, are the sulphates and selenates of the alkali metals potassium, rubidium, and caesium, which crystallise in the rhombic system of symmetry; and thirty members of the well-known monoclinic series of double sulphates and selenates, containing one of the three alkali-metals just mentioned, another metal of the magnesium or iron type, and six molecules of water of crystallisation.

The research had two main objects. First, to discover the effect of replacing one alkali-metal by another; and second, the effect of replacing the lighter element, sulphur, contained in the acid radicle of the salt, by the heavier element, selenium. The three metals, potassium, rubidium, and caesium, belong strictly to the same family of chemical elements, according to the now famous periodic classification of Newlands and Mendeleeff. They are the most electro-positive metals known, and the weights of their atoms are related in a most interesting manner, that of rubidium being the mean of the atomic weights of potassium and caesium. It was, therefore, to be expected that any difference of form or properties brought about by the replacement of any one of these metals by another would be as great as could ever be produced by a change of this kind, and it might be hoped would be adequately great to enable a true idea of its character to be obtained. The very fact that no differences in the angles of the crystals of so-called isomorphous salts had, up to the commencement of this work, been detected with certainty, shows how very small, at most, such differences must be. It will also be evident that for such a research only the most perfectly formed and homogeneous of crystals must be employed. It is a primary essential that the faces of the crystals shall yield perfect images of the signal-slit of the goniometer, and in order that this may be so they must be absolutely plain surfaces. Indeed, if one may be forgiven a slight equivocation in the spelling of a word, the ladies present may be interested to hear that the beauty of crystals lies in the planeness of their faces.

The mode of measuring the angles between crystal faces on the goniometer, and at the same time the difficulty offered by imperfect faces, may be illustrated with the aid of this large crystal of quartz. It is mounted upon one of the actual goniometers employed in the work, but the size of the crystal is enormously greater than those actually used in the research, which rarely exceeded the size of a pin's head. Such small crystals are much more free from distortion than larger ones.

You now see on the screen the image of the goniometer signal-slit reflected from one of the faces of the quartz crystal. This image is an irreproachable one, the face reflecting it being truly plane. Its narrow part is capable of accurate adjustment to a vertical cross-wire. On rotating the crystal so as to bring the image from the next face, of the particular zone which has been previously adjusted, into the field, you observe that this also is a

very good image, but much weaker than the other. This is simply because it is derived from a much smaller face, but the face is quite plane. The next image you see is a bad one, being not only a multiple image, but distorted. Such an image would be quite useless for our purpose. Thus, on rotating round the whole zone—for it is one of the geometrical properties of crystals that the faces lie in zones—we find that some images are good and some are bad. In the case of very small crystals it generally happens that the greater number of images afforded by the faces are either one thing or the other, and crystals must be selected yielding the maximum of good images.

The first result of importance that has been brought to light is that, with regard to each series of salts, there is an interesting relationship between the general exterior character—or habit, as the crystallographer terms it—of any triplet of salts containing potassium, rubidium, and caesium respectively.

We will see on the screen, for instance, the configurations of the three double salts, potassium zinc sulphate, rubidium zinc sulphate, and caesium zinc sulphate. The same planes are present in all, but very differently developed. In the potassium salt the shape is determined by the large development of the prism zone (β faces), and the large flat end faces of the basal plane, c . The caesium salt, on the other hand, exhibits a prismatic habit formed by the faces of the clinodome, q , and the basal plane c is reduced to a strip. Intermediate between these two types comes the rubidium salt, and this is the case with the rubidium salt of every one of the sixteen triplets examined.

Hence the habit clearly follows the order of the atomic weights of the alkali metals.

The next general result arrived at is, that small angular differences between the faces have been established, but they rarely amount to a degree in magnitude. And what is of even more interest is, that the faces of every rubidium salt are inclined to each other at intermediate angles to those of the potassium and caesium salts.

This important fact may be illustrated by the vacuum-tube model on the table, which has been specially constructed to emphasise the point. The three crystal outlines represent sections through one of the principal planes of the alkaline sulphates or selenates. The outer one represents the outline of caesium sulphate or selenate, the middle one that of the rubidium salt, and the inner one a section of the potassium salt. The intermediate inclination of the domal faces will be clearly apparent. This is only one of some sixty angles which have been examined, and all show the same beautiful progression in the order of the atomic weights.

In the case of the principal angle of the monoclinic series, which determines the inclination of the inclined axis characteristic of that system of symmetry, the change of angle is directly proportional to the change in atomic weight.

Before concluding what may be said about the exterior morphology, reference may be made to one other fact, which follows from that just referred to and from the results of careful determinations of the specific gravity of the salts. The latter enables one to arrive at the molecular volume, which in the case of salts belonging to the same series does actually represent the relative volume of the chemical molecules. The size of the molecules of the rubidium salt was in every case found to be intermediate between the sizes of the molecules of the potassium and caesium salts. By combining these molecular volumes with the lengths of the crystallographical axes as afforded by the angular measurements, it has been possible to determine the relative dimensions, in the three directions of space, of each chemical molecule.

This was preceded by a proof, which has been confirmed by Dr. Fock, of Berlin, from an entirely different point of view, that the unit of the crystal structure in these salts was identical with the chemical molecule, and was not an aggregate of such,

The result has been to show that the sizes, or at any rate the distances apart from centre to centre, of the chemical molecules in the three rectangular directions of space are intermediate in the case of every rubidium salt. Thus the directional dimensions of the molecules, like the angles of the crystal structure, follow the order of the atomic weights of the alkali metals.

This fact is clearly exhibited in exaggerated fashion by the model, and it will be observed that the intermediate position of the rubidium salt is somewhat nearer to that of the potassium salt than to that of the caesium salt.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, Wednesday, June 18th, 1902.

Prof. EMERSON REYNOLDS, Sc.D., V.P.R.S., President, in the Chair.

MESSRS. Russell, Lattey, Ionides, Haas, and Heaton were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Guy Dunstan Ricketts, Sir John Cass Technical Institute, E.C.; Edgar Stansfield, B.Sc., Technical College, Sanderland; Thomas Edward Wallis, 78, Essex Road, Islington, N.

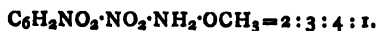
The PRESIDENT announced that the Council that afternoon had decided that the Ordinary Meetings of the Society for the ensuing session should be held, as far as possible, alternately, on Wednesdays at 5.30 p.m., and Thursdays at 8 p.m.

A ballot for the election of Fellows was held, and the following were subsequently declared duly elected:—James Handby Ball, B.Sc.; Edwin Thomas Holman Bucknell; Bryce Cudleigh Burt, B.Sc.; Arthur Crozier Claudet; William Thomas Clough; Henry Wilson Davis; Percy J. Ferris; Edgar William Foll; Francis E. Francis, B.Sc., Ph.D.; John Longsdon Garle; Alexander Gow, B.Sc.; Thomas B. Hallowell; Archie Cecil Osborn Hann; Walter Ernest Harrison; George Herbert Leader, B.Sc.; Charles B. Lessner; James Butler Moody; Thomas Henry Moore; Sinnott Valentine O'Connor; Percy Philip Phillips, Ph.D.; George Paton Pollitt, Ph.D.; William E. F. Powney; Herbert Purton; Stephen Jamieson Ralph; Edwin Ralphs; Prafulla Chandra Ray, D.Sc.; Harold James Roast; William Pearson Skeritchly; William C. S. Stanger; Hector Stewart; John William Wells; Joseph West; Edward J. Wheeler.

Of the following papers, those marked * were read:—

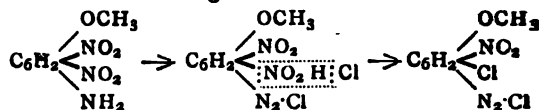
*106. "Elimination of a Nitro-group on Diazotisation. Dinitro-*p*-anisidine." By R. MELDOLA and J. V. EYRE.

Dinitro-*p*-anisidine, obtained by nitrating acet-*p*-anisidine and hydrolysing the acetyl derivative, was shown by the authors to have the constitution—

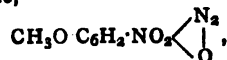


This compound, on diazotisation in acetic acid in presence of sulphuric or nitric acid, behaves normally, and forms a dinitrodiazonium salt, which on heating with alcohol gives dinitroanisole, $\text{C}_6\text{H}_3\cdot\text{NO}_2\cdot\text{NO}_2\cdot\text{OCH}_3 = 2:3:1$, of m. p. 110°. The dinitrodiazonium salts combine with alkaline β -naphthol in the ordinary way to form the azo compound, $\text{CH}_3\text{O}\cdot\text{C}_6\text{H}_2(\text{NO}_2)_2\cdot\text{N}_2\cdot\text{C}_{10}\text{H}_6\cdot\text{OH}\beta$. When diazotised in the presence of hydrochloric or acetic acid, one of the nitro-groups is eliminated as in the case of dinitro-*o*-anisidine (*Trans.*, 1900, lxxvii., 1172; *Proc.*, 1901, xvii., 131; *Trans.*, 1901, lxxix., 1076). The authors showed that it is the 3-nitro group, that is, the group which is *ortho* with respect to the diazonium group, which is thus eliminated. The product, when hydrochloric acid

is used, contains chlorine in place of the 3-nitro-group, and is formed according to the scheme:—



No diazoxide appears to be formed in this case, but the replacement of the nitro-group by chlorine takes place at once at the ordinary temperature. The chloronitrodiazonium salt, when boiled with alcohol, with or without the addition of alkali, gives chloronitroanisole, $\text{C}_6\text{H}_3\cdot\text{Cl}\cdot\text{NO}_2\cdot\text{OCH}_3 = 3:2:1$. The same diazonium salt, on combination with alkaline β -naphthol, gives the azo-compound, $\text{CH}_3\text{O}\cdot\text{C}_6\text{H}_2\text{Cl}\cdot\text{NO}_2\cdot\text{N}_2\cdot\text{C}_{10}\text{H}_6\cdot\text{OH}\beta$. When diazotised in the presence of acetic acid alone, the NO_2 group is replaced by hydroxyl, and the diazonium salt combines with β -naphthol to form the hydroxyazo-compound, $\text{CH}_3\text{O}\cdot\text{C}_6\text{H}_2\cdot\text{NO}_2\cdot\text{OH}\cdot\text{N}_2\cdot\text{C}_{10}\text{H}_6\cdot\text{OH}\beta$. In this case, a diazoxide,—



may be formed as an intermediate product, but this has not yet been isolated.

The displacing of the nitro-group in the two dinitroanisidines now investigated tends to show that the elimination of this group follows the ortho-para law, and experiments for the purpose of testing this conclusion will be undertaken in due course.

*107. "Preliminary Notice of some New Derivatives of Pinene and other Terpenes." By W. A. TILDEN and H. BURROWS.

Pinene nitroschloride is remarkable as being the only known compound containing the elements of pinene from which that hydrocarbon may be regenerated. The addition of one molecule of hydrogen chloride to pinene gives the familiar monohydrochloride (artificial camphor, m. p. 128°), from which, by withdrawal of hydrogen chloride, camphene, and not pinene, results. The addition of two molecules of hydrogen chloride to pinene gives rise to dipentene dihydrochloride (m. p. 50°), from which, by removal of two molecules of hydrogen chloride, a mixture of liquid isomerides of pinene is obtained. On the other hand, when pinene combines with nitrosylchloride, a compound is formed which contains a nitroso-group. This is indicated by the fact that it is capable of reacting with aniline to yield a diazotised product, leaving a hydrocarbon which is apparently identical with pinene, though optically inactive (Wallach, *Ann.*, 1889, cclii., 132; 1890, ccviii., 344).

The authors have repeated this experiment, and although the reaction is not so simple as they were led to infer from Wallach's account of it, they have verified the fact that the recovered hydrocarbon combines readily with nitrosylchloride, forming a nitroschloride which melts at 110°, and from which a benzylnitrolamine melting at 125° was obtained. The corresponding compounds formed from ordinary dextro-pinene melt at 103° and 122–123° respectively (Wallach). The fact that this recovered pinene is optically inactive but not resolvable into two optical isomerides has not attracted the attention it deserves. If optically inactive pinene really possesses the same constitution as active pinene, this would imply the existence in the molecule of two asymmetric carbon atoms similarly combined. The formulæ given by Wallach, Wagner, and other chemists are therefore inadequate to explain the facts.

In the hope of getting some new light on the problem, attempts have been made to produce new derivatives of the hydrocarbon by adding carbon in various forms to the unsaturated part of the molecule, with the idea of obtaining acids or other compounds of determinable constitution. Several methods have been tested in a pre-

liminary way, but at present only one has met with success.

When pinene nitroschloride is gently heated with an equivalent quantity of potassium cyanide in the presence of 90 per cent alcohol, a compound having the composition of pinene nitrosocyanide is formed amounting to about 40 per cent of the nitroschloride employed. The viscous by-products have not been completely examined. The cyanide is a crystalline compound which forms large prisms, of which the melting-point is 171° . On analysis, the compound gave 14.47 per cent of nitrogen, $C_{10}H_{16}NOCN$, requiring 14.58 per cent. It dissolves readily in alcohol, ether, acetic acid, and toluene, but very sparingly in light petroleum, and it is practically insoluble in water.

The constitution of this nitrosocyanide is at present an unsettled question. It is unaltered by boiling with 30 per cent alcoholic potash for several hours. On the other hand, it is completely destroyed with formation of uncrystallisable products when heated with dilute sulphuric acid or with hydrochloric acid, either in alcoholic solution or in glacial acetic acid at 100° . It is therefore doubtful whether the compound is a nitrile. There are, however, other cases on record of nitriles which resist the action of alkalis, and the question is still under investigation.

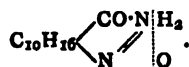
By digesting the cyanide with alcoholic potash and methyl iodide, a methyl derivative is produced which is distinguished by the readiness with which it forms large, colourless, prismatic crystals. The melting-point of this compound is 67° , and it contains 13.64 per cent of nitrogen, $C_{10}H_{15}CH_3 \cdot NO \cdot CN$ requiring 13.60 per cent.

The action of reducing agents on the cyanide is rather remarkable. Three methods were successively employed, namely, (1) the action of sodium on a boiling solution of the cyanide in amyl alcohol, (2) the action of sodium on a boiling solution in ethyl alcohol, and (3) the action of zinc in the presence of alcohol and hydrochloric acid. In the reductions with sodium, some ammonia was evolved, and a cyanide was produced in considerable quantity, together with an almost quantitative yield of pinylamine.

The acid reducing agent produced practically no change, but when zinc dust and an alkaline solution were employed ammonia was evolved, and on steam distillation an oil of camphoraceous odour passed over.

A small quantity of a second crystalline, but not basic, compound, not yet investigated, was produced in both cases, and was extracted from the alkaline residue.

Pinene nitrosocyanide dissolves in concentrated sulphuric acid without coloration but with evolution of considerable heat. The solution, when diluted with water, yields the unchanged nitrosocyanide, but when the solution in sulphuric acid is digested at about 100° for an hour or two, the addition of water does not cause an immediate precipitate. On allowing the liquid to stand, a new compound is deposited in tufts of crystals, which, after re-crystallisation from alcohol, appear in the form of well defined plates which melt and decompose at about 220° . It is soluble in aqueous potash, and, somewhat strangely, also in hydrochloric acid. This compound contains 14.38 per cent of nitrogen, which corresponds closely with the amount required either by a polymerised product, $(C_{10}H_{16} \cdot NO \cdot CN)_n$, or by the product which would result from the formation of the corresponding acid amide and the subsequent elimination of water, thus:—



This is, however, a subject for further investigation.

Pinene nitrosocyanide, placed in contact with warm nitric acid (sp. gr. 1.4), liquefies but does not dissolve, while a small quantity of nitrous gas is evolved. In a few minutes, the reaction seems to be complete, effervescence ceases, and on pouring the whole into cold water, a solid is precipitated which crystallises readily from alcohol in colourless prisms. This compound is insoluble in aqueous

alkali. It melts with decomposition at 105° . Analysis gave 16.30 per cent of nitrogen. The formula of a mononitrosocyanide, $C_{10}H_{16} \cdot NO_2 \cdot CN$, requires 13.46, while that of a dinitrosocyanide, $C_{10}H_{15} \cdot (NO_2)_2 \cdot CN$, requires 16.60 per cent of nitrogen. It is probable, therefore, that the nitroso-group is oxidised, and nitration occurs at the same time.

An attempt to produce a nitrosocyanide from limonene nitroschloride gave a liquid product which is awaiting further investigation. Terpinene nitrosate is also expected to yield corresponding products.

With regard to the hydrochlorides obtainable from pinene, the monohydrochloride (m. p. 128°) refused to react under any circumstances with either cyanide of potassium or of silver. An alcoholic solution of dipentene dihydrochloride digested with potassium cyanide in the cold for fourteen days gave the liquid monohydrochloride, $C_{10}H_{16} \cdot HCl$, and the latter by continuing the action of the cyanide at about 120° , gave very pure terpinene, which was identified by its boiling-point (181°) and by the production of the characteristic crystalline nitrosate.

*108. "The Colour Changes Exhibited by the Chlorides of Cobalt and some other Metals from the Standpoint of the Theory of Electro-affinity." By F. G. DONNAN and H. BASSETT, jun.

The colour changes exhibited by aqueous and alcoholic solutions of the chlorides of cobalt, copper, and ferric iron on variation of temperature or dilution, or on the addition of various chlorides, such as those of hydrogen, calcium, magnesium, zinc, mercury, are shown to be largely due to the formation or dissociation of complex anions containing a metallic atom in association with chlorine. Evidence was brought forward to show that the dehydration theory is inadequate to explain the observed phenomena. Experiments on the motion of boundaries between different liquid layers on the passage of an electric current point to the existence of complex negative ions. Thus, in the case of cobalt chloride, the blue solutions travel towards the anode, the red solutions towards the cathode. Further experiments on the combination between cobalt and other chlorides in aqueous and alcoholic solutions admit of explanation on the above hypothesis.

*109. "The Stereochemical Formula of Benzene." By J. E. MARSH.

The author discussed the objections to the stereocentric formulæ of benzene brought forward by Graebe (*Ber.*, 1902, xxxv., 526), and pointed out that, according to the latter, rings identical in structure, which their properties do not admit of, would be assigned to benzene and naphthalene. The author considers that all the eight stereocentric formulæ are possible for benzene and its derivatives; that orthophthalic acid would have the *trans*-centric form, in which case it could form an anhydride, though the meta-acid could not, since the hexahydro-ortho-acid alone forms an anhydride in the *trans*-form. Thus, proximity of groups alone would not account for the formation of anhydrides, nor of carbonates and methylene ethers of dihydric phenols. Further, the absence of optically active compounds is accounted for if we regard the Kekulé formula as a labile form of the *cis*-centric, for the Kekulé model can close in in two ways to form *cis*-centric formulæ, one being the optical image of the other.

With regard to substitution, those compounds which yield meta-derivatives contain an oxidisable group, whilst those which yield ortho- and para-derivatives contain a reducible group. In the former case, the repulsion of hydrogen leads to the *trans*-centric form with production of meta-derivatives. In the latter case, the attraction of hydrogen to the group leads to *cis*-centric hydrogen in the ortho- and para-positions. Bamberger's dicentric formula for naphthalene was also criticised, as it appears stereochemically impossible, and is not in accord with the constitution of allied hydrocarbons.

*110. "An Accurate Method of Determining the Compressibility of Vapours." By B. D. STEELE, D.Sc.

In the course of an investigation with Prof. Ramsay, it became necessary to determine, with a high degree of accuracy, the compressibility of certain vapours at low pressures. The apparatus employed consists of a combination of volume apparatus and pressure gauge, so constructed as to be capable of being contained together in a glass jacket which can be maintained at a constant temperature. The pressure is measured by setting two surfaces of mercury to carefully ground glass points the difference in level between which is known. The volume is obtained by weighing the mercury entering or leaving the apparatus.

Experiments with the vapours of benzene and ether have been made in the apparatus, and curves obtained showing the variation of PV with P for pressures varying from 40 to 200 m.m.

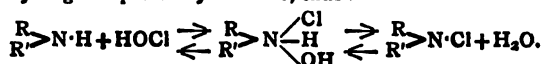
*111. "A New Type of Substituted Nitrogen Chlorides." By F. D. CHATTAWAY.

No substituted nitrogen chlorides known up to the present have three negative groups attached to the nitrogen. All contain at least one hydrocarbon residue.

Such a series of compounds of the type—



where R and R' are acyl groups, is yielded by the diacyl amides. These, when treated with hypochlorous acid or, under certain conditions, with chlorine, have their imino-hydrogen replaced by chlorine, thus:—



The diacyl nitrogen chlorides are colourless, well-crystallised compounds which show the typical behaviour of substances having halogen attached to nitrogen.

Dibenzoyl Nitrogen Chloride, $\begin{matrix} C_6H_5 \cdot CO \\ C_6H_5 \cdot CO \end{matrix} > N-Cl$.—In order

to obtain this compound, dibenzamide, prepared by the action of fuming sulphuric acid and phosphorus pentoxide on benzonitrile, was dissolved in acetic acid. To the solution, an excess of a strong solution of bleaching powder was added. Chlorine was vigorously evolved, and a yellow oil separated. Chloroform was added, and the whole well shaken for a few minutes. The chloroform solution was then run off, washed with water, and finally with a dilute solution of potassium bicarbonate, dried over fused calcium chloride, and the solvent evaporated at a low temperature in a rapid current of dry air. The nitrogen chloride crystallised out as a white solid (m. p. 89°), readily soluble in chloroform and in benzene, but sparingly in petroleum. It crystallises from a mixture of chloroform and petroleum (b. p. 60–80°) in colourless, six-sided prisms with domed ends. It was analysed in the usual way, by adding an excess of hydriodic acid to a weighed quantity dissolved in chloroform, and estimating the liberated iodine by a standard solution of sodium thiosulphate:—

0.2446 liberated I = 18.7 c.c. N/10 I. Cl as :N:Cl = 13.55.
 $C_{12}H_{10}O_2:N$ Cl requires Cl as :N:Cl = 13.65 per cent.

When heated with water or dilute acids it is hydrolysed, hypochlorous acid being liberated and dibenzamide reformed, the latter undergoing further hydrolysis to benzoic acid and benzamide, and finally, if the heating be prolonged, to benzoic acid and ammonia.

Di-p-toluyyl Nitrogen Chloride, $\begin{matrix} CH_3 \cdot C_6H_4 \cdot CO \\ CH_3 \cdot C_6H_4 \cdot CO \end{matrix} > N-Cl$.—

This was prepared from di-p-toluylamide by the method previously described. It closely resembles the dibenzoyl derivative in properties and in solubility. It crystallises in long, colourless, six-sided prisms with domed ends (m. p. 129°).

0.2121 liberated I = 14.8 c.c. N/10 I. Cl as :N:Cl = 12.37.
 $C_{16}H_{14}O_2:N$ Cl requires Cl as :N:Cl = 12.32 per cent.

The author desires to reserve the further investigation of these and similar compounds.

*112. "The Preparation of Pure Chlorine and its behaviour towards Hydrogen." By J. W. MELLOR and E. J. RUSSELL.

A quantity of carefully dried and purified silver chloride was placed in a V-shaped tube of the hardest Jena glass, in which were also inserted, as electrodes, stout carbon rods specially prepared for the electrolysis of fused salts. An air-tight joint having been made, the salt was fused and a current of 2.8 amperes passed through. Chlorine was evolved in a steady stream. Foreign gases and moisture were removed by occasional reversal of the current, and repeated exhaustion with an automatic Sprengel pump.

The silver tree was destroyed by raising the temperature and increasing the current to 5 amperes. The tree then either melted or was shattered; electrolysis was discontinued until the former temperature was restored; when started again, it was found to proceed in a normal manner.

Hydrogen was prepared by the action of steam on sodium, and was absorbed by palladium. The whole apparatus was then exhausted, the palladium heated, and hydrogen evolved.

The gases were sealed up in the inner and outer bulbs respectively of the condensers now largely used with Soxhlet extractors. Each bulb contained a quantity of carefully purified phosphorus pentoxide, the inner bulb containing also a small piece of glass rod. After drying for some months in the dark, the vessels were shaken to break the inner bulb, and the gases mixed by diffusion. Only condensers which had approximately equal capacities for the inner and outer bulbs were used; hydrogen was put in at the same pressure as the chlorine, so that the volumes of the two gases were nearly equal.

A small electric spark passed through a mixture so obtained caused an explosion, and combination was found to have been practically complete. Apparently drying does not affect the action of the spark.

Mixtures of moist hydrogen and chlorine in similar bulbs were found to explode at about 260°. One of the bulbs containing the purified mixture was therefore heated to 270° for some minutes, but no explosion took place. On opening the bulb, it was found that there had been practically no combination. Another bulb was heated for ten minutes to 450°. Still no explosion occurred, but about 80 per cent of the mixture had combined. Some of the phosphorus pentoxide had volatilised, and as it was found impossible to heat the gases without also heating the pentoxide, it is still uncertain whether this slow combination is a direct action or a surface action. Another bulb was exposed to bright sunshine at Wye for three days, but no explosion took place; and only about 30 per cent of the hydrogen and chlorine had combined.

(To be continued).

A New Reaction for Cholesteroline.—L. A. Tchougaef. —The chlorides of the fatty or aromatic acids when heated with a little cholesteroline in solution in chloroform give characteristic colourations in the presence of $ZnCl_2$. To detect cholesteroline in a practical manner it is best to operate in the following manner:—The cholesteroline is dissolved in anhydrous acetic acid; to the solution we add an excess of chloride of acetyl and a few pieces of chloride of zinc; the colour appears on heating, and attains its maximum intensity after boiling for five minutes over a naked flame; the solution becomes red or rose-coloured according to the amount of cholesteroline present, and it also has a greenish-yellow fluorescent appearance. This reaction is much more sensitive than that of Liebermann, as the colour is still apparent with a dilution of 1 part of cholesteroline in 80,000 parts of liquid.—*Journ. Soc. Phys. Chim. R.*, vol. xxxii., p. 363.

CORRESPONDENCE.

THE PATENT LAW AMENDMENT BILL.

To the Editor of the Chemical News.

SIR,—Among the modifications proposed by Mr. Gerald Balfour to be introduced into his Patent Law Amendment Bill, when he moved the second reading on Friday last, was one the effect of which is probably not generally understood, but which, if adopted, will certainly go far to deprive the A& of practical value, and will most probably render it entirely abortive. I refer to the proposal to remove the jurisdiction in respect of Compulsory Licenses from the High Court and to confer it upon the Judicial Committee of the Privy Council.

The Judicial Committee consists of very eminent lawyers, and commands in consequence a large measure of respect. Its decisions on points of principle would be accepted by the whole community with the utmost confidence; but for the particular subject-matter which it is proposed to refer by this A& to that tribunal, the tribunal is most unsuitable. I will mention only two of its disqualifications.

First, it is the most expensive tribunal in the Kingdom. Costs at the Privy Council are always on the "higher scale"; that is to say, on a scale even higher than what in the High Court is known as the "higher scale" and allowed there only in exceptional cases. As an illustration of the lavish expenditure in costs incidental to proceedings before the Judicial Committee, I may mention that Counsel's fees for a consultation, which in a case before the High Court would amount to three guineas, would, if the same consultation were held in connection with litigation before the Privy Council, amount to ten guineas. These extravagant costs will alone be fatal to the relief proposed by the Bill.

Next, the Judicial Committee has no subordinate judicial staff to whom questions of detail can be referred. In Appeals—and the Judicial Committee was organised to act as an Appellate Court—this does not matter. The Appeal Court decides the point in question and remits the working out of its decision in detail to the lower Court. In this way accounts can be adjusted and all the administrative detail worked out by the machinery of subordinate tribunals organised upon a different plan for the conduct of such business. But when the Judicial Committee is directed to undertake the work of a Court of First Instance this deficiency of a staff of referees becomes a serious defect. It has proved a serious hindrance to the work of the Judicial Committee in other matters of a like kind, and it will certainly cripple the tribunal for the present purpose. It would be absurd to expect the Lords of Appeal to spend hours and days in hearing evidence and argument as to whether a royalty should be at 10 per cent on selling price or 15 per cent on cost price, or, say, a penny a pound. A question of that sort can only be effectually investigated by a referee. Four or five of the most distinguished Judges in the land ought not to be asked to sit in conclave on such a question—and will not do it, whatever the A& of Parliament may say. A Judge of the High Court would not do it: but he would get over the difficulty by sending that part of the enquiry, as he already does the examination of intricate accounts, to an official or a special referee.

These, then, are two of the inconveniences involved in the alteration now proposed; and even if they were all, they would, I submit, be sufficient to justify the strongest possible protest on the part of those interested in the success of our Patent Law against the ill-considered proposal to substitute the Judicial Committee for the High Court as the tribunal to decide on questions of compulsory licenses.—I am, &c.,

J. W. G.

Temple, July 10, 1902.

THE MELTING-POINT OF CHROMIUM.

To the Editor of the Chemical News.

SIR,—With reference to the note by Mr. E. A. Lewis on "The Melting-point of Chromium" (CHEMICAL NEWS, vol. lxxxvi., p. 13), it may be as well to point out that as the specimen he conducted his experiments upon contains 1 per cent of impurities, consisting mostly of aluminium (an element reputed to lower the melting-point of certain metals enormously), it is possible that Mr. Lewis's determination is still faulty as that of the melting-point of the pure metal.

In some careful experiments by Osmond, the melting-point of mild steel (*i.e.*, iron plus at least 0.5 per cent of other matters) was found to be 1475° C., and that of hard steel (iron containing 1.0 per cent other matters) 1410° C. This is a difference of 65° for an addition of 0.5 per cent more of other matters.

As aluminium has probably a very considerable influence upon the melting-point of chromium, it would appear that Mr. Lewis's determination is only of value as a record of the melting-point of the particular alloy used by him, and as such it would be interesting to know the precise nature of the 1 per cent other matters present in it.

Osmond gives the melting-point of a chrome alloy containing 80 per cent of chromium as 1485° C.; this is only 35° below that now found by Mr. Lewis in an alloy containing 19 per cent more chromium. I think it may be fairly implied from the opening sentence of Mr. Lewis's note that it is suggested that the melting-point of pure chromium has now been obtained.—I am, &c.,

T. W. Hogg.

Newburn Steel Works, July 14, 1902.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxiv., No. 25, June 23, 1902.

Researches on the Reciprocal Action of two Liquids.—M. Berthelot.—The author examines the action of electric piles formed of two liquids, and finds that currents of measurable intensity are produced. He also finds that there is a development of a true electromotive force capable of attaining a specific and definite limit for each of the systems examined, and which is analogous to Volta's primitive pile. He then determines the intensity of the current produced by such piles, and makes a comparison between the intensity of these and those in which the products of electrolysis are not dissipated by the phenomena of diffusion, polarisation, &c.

Chlorating Properties of a Mixture of Hydrochloric Acid and Oxygen.—Camille Matignon.—Tellurium, gold, and platinum, in all their different forms, are attacked by a mixture of hydrochloric acid and oxygen at temperatures much below that of the oxidation of hydrochloric acid gas in oxygen. This mixture, as can be predicted from theoretical considerations, constitutes a chlorating agent of a great number of materials. In the three cases mentioned, its action can be compared to that of free chlorine.

Acidity of Pyrophosphoric Acid.—H. Giran.—Thomsen determined the heats of neutralisation of a molecule of pyrophosphoric acid successively with one, two, and four molecules of soda. The author now completes the investigation, and to obtain more comparable results he prepares the solid acid and the four anhydrous

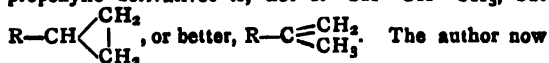
sodium salts. He obtains the following numbers, on the assumption that—

Na sol. + aq. = NaOH diss.	+42.4
P ₂ O ₅ H ₄ sol. + Na sol. = P ₂ O ₅ NaH ₃ sol. . .	+64.80
P ₂ O ₅ NaH ₃ sol. + Na sol. = P ₂ O ₅ Na ₂ H ₂ sol. . .	+59.90
P ₂ O ₅ Na ₂ H ₂ sol. + Na sol. = P ₂ O ₅ Na ₃ H sol. . .	+46.26
P ₂ O ₅ Na ₃ H sol. + Na sol. = P ₂ O ₅ Na ₄ sol. . .	+45.18

He therefore finds that pyrophosphoric acid is a tetrabasic acid possessing four strongly acid functions identical with one another.

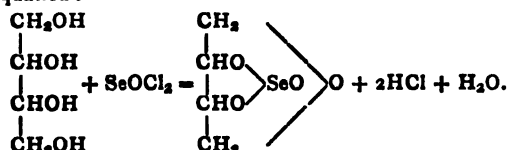
Displacement of Strong Bases by Ammoniacal Cupric Oxide.—M. Bouzat.—The complex base contained in ammoniacal solutions of cupric hydrate is a very strong one. It displaces almost integrally ammonia and its salts. It is nearly as strong as potassium, and in concentrated and strongly ammoniacal solution it precipitates chalk to a very large extent.

Phenylic Migration of Phenylethylene and its Derivatives.—M. Tiffeneau.—M. Bougault showed that the iodohydrines of the derivatives R-CH₂-CH=CH₂ are not decomposed either by HgO or by AgNO₃, whilst iodohydrines of the propenylic derivatives, R-CH=CH-CH₂, are transformed either by HgO or AgNO₃ into aldehydes, R-CH(CH₃)-CHO; and as, on the other hand, iodohydrine of styrolene gives, under the same conditions, phenylacetic aldehyde, the author concludes that the structure of the initial propenylic derivatives is, not R-CH=CH-CH₂, but



The author now further examines the mechanism of these reactions.

Action of Selenyl Chloride on Erythrite.—C. Chabré and R. Jacob.—The authors find that the dehydration which erythrite undergoes under the action of selenyl chloride causes it to become a primary alcohol group, and the fixation of selenium takes place in the secondary alcohol groups. The constitution of the product formed, and the reaction causing it, is shown by the following equation:—



Dibenzoylhydrazobenzene.—P. Freundler.—The author repeats the experiments of Biehringer and Busch, and establishes the following reaction:—When benzoic anhydride acts on hydrazobenzene in presence of absolute alcohol at 120° in a sealed vessel, azobenzene is produced, and also a white substance which melts at 161°. This product possesses the properties and composition as described by Biehringer and Busch, but the author finds it to be simply benzanilide.

Acyl Derivatives of Isopyromucic Acid, Acetate, Benzoate, and Pyromucate of Isopyromucyl.—G. Chavanne.—For the purpose of verifying the existence of the phenolic or enolic function of isopyromucic acid, the author examines the action of acid chlorides on this compound. He thus prepares the acetate, benzoate, and pyromucate of isopyromucyl.

MISCELLANEOUS.

Saponification of Nitrate of Ethyl by Water.—E. Biron.—To determine the speed of saponification of nitrate of ethyl by water, the author kept a mixture of water and ether at a constant temperature; the latter must be in excess, so that during the whole of the operation the aqueous solution of the ether remains saturated,

and the concentration is constant; at determined intervals samples of the solution are taken in which the amount of HNO₃ is titrated by means of Ba(OH)₂ in the presence of phthalein. We find that at first the solubility of the nitrate of ethyl in 100 grms. of water (between 55° and 85°) is 2.239-0.03642t+0.0003152t². The experiment shows that the speed of saponification increases in the same ratio as the amount of nitric acid contained in the water, and can be expressed as follows:—

$$\frac{dx}{d\theta} = (k_1 + k_2x)C;$$

x is the amount of ether decomposed, θ the time in hours, C the concentration of the ether, k the constant of the reaction with water, k_2 the constant of catalytic acceleration due to the presence of nitric acid. At the temperature of 70°, $C = 0.1481$; $k = 0.004246$; $k_2 = 0.0135$. We see that the normal nitric acid solution saponifies about 4.17 times quicker than pure water.—*Journ. Soc. Phys. Chim. R.*, vol. xxxii., p. 636.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

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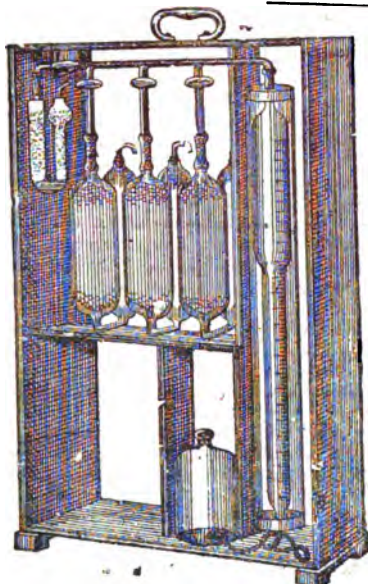
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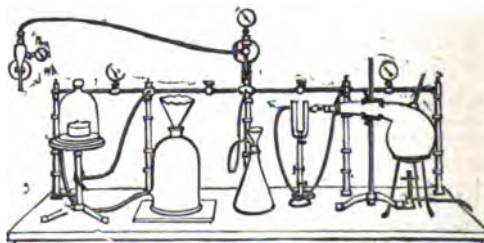
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Vol. LXXXVI., No. 2226.

NINTH ANNUAL REPORT OF THE COMMITTEE ON ATOMIC WEIGHTS. DETERMINATIONS PUBLISHED IN 1901.*

By F. W. CLARKE.

(Concluded from p. 26).

Tellurium.

AN interesting contribution to our knowledge of tellurium is due to Steiner (*Berichte*, xxiv., 570), who has measured its atomic weight by partial analyses of diphenyl telluride. In this compound, by combustion, the carbon was determined, and from that determination the atomic weight of the metal was computed. The data, for two separate fractions of material, are as follows:—

Weight $C_{12}H_{10}Te$.	Weight CO_2 .	Per cent C.	Atomic weight.
1. { 0.2295	0.5512	51.39	126.1
{ 0.2559	0.4811	51.28	126.7
{ 0.23065	0.4341	51.34	126.4
2. { 0.2140	0.4031	51.38	126.2
{ 0.2578	0.4849	51.31	126.6

Mean 126.4

Calculated with $C=12.003$, $H=1.008$. The results are, of course, only approximate, and are intended by the author as an indication of a promising method, and as evidence that the atomic weight of tellurium falls below that of iodine, as demanded by the Periodic Law.

Steiner also gives two analyses of diphenyl selenide, with determinations of the atomic weight of selenium based upon them. The data are:—

Weight $C_{12}H_{10}Se$.	Weight CO_2 .	Per cent C.	Atomic weight.
0.2812	0.6375	61.85	78.8
0.5371	1.2158	61.71	79.3

Another communication upon the atomic weight of tellurium is by Pellini (*Berichte*, xxiv., 3807), who employed the old methods of oxidation and reduction connecting Te with TeO_2 . His material, however, was purified by means of two organic compounds, diphenyl telluride and diphenyl tellurium dibromide, from which the metal used in the determinations was finally obtained.

First, Te was oxidised to TeO_2 by means of nitric acid. In the first three determinations the dioxide was heated to 400° ; in the remaining four it was fused. The sixth experiment is only partially stated; the figures in the third and fourth columns have been computed by your Committee. The final mean result ($O=16$) is that given by Pellini.

Weight Te .	Weight TeO_2 .	Per cent Te in TeO_2 .	Atomic weight.
1.0679	1.3353	79.968	127.70
1.5469	1.9354	79.926	127.41
2.2386	2.7980	80.007	128.05
2.4522	3.0665	79.967	127.73
2.0977	2.6239	79.945	127.56
2.0442	2.5575	79.929	127.43
2.0434	2.5556	79.957	127.66

Mean 127.65

The reduction experiments, performed in hydrogen by Staudenmayer's method, were only three in number, as follows:—

Weight TeO_2 .	Loss of weight.	Per cent Te in TeO_2 .	Atomic weight.
1.4680	0.2944	79.945	127.56
1.9968	0.3993	80.000	128.02
1.9575	0.3932	79.913	127.30

Mean 127.62

The figures obtained are not remarkably concordant, but they tend to confirm the older determinations, and to place tellurium above iodine.

Still a third memoir upon this atomic weight is due to Koethner (*Liebig's Ann. Chem.*, cccix., 1), whose work is more than ordinarily elaborate and thorough. His material was scrupulously purified, and studied spectroscopically; and the only (minute) impurities which seem to be possibly present are such as would tend to lower the observed atomic weight. Several methods of determination were attempted, but the final results rest upon the study of the basic tellurium nitrate, $Te_2O_3 \cdot NO_3 \cdot OH$. This salt, on heating under proper precautions, is reduced to TeO_2 . In the second series of determinations, as given below, the tellurium which served as the starting-point had been distilled *in vacuo*; in the first series that precaution was not taken. The weights were not reduced to a vacuum, and the calculations were based upon $O=15.88$, $N=14.93$, and $H=1$.

SERIES I.

Weight of salt.	Weight TeO_2 .	Atomic weight Te .
2.9373	2.4522	126.39
2.7982	2.3361	126.40
2.8554	2.3840	126.46

Mean 126.41
If $O=16$, Te =127.36

SERIES II.

Weight of salt.	Weight TeO_2 .	Atomic weight Te .
5.30270	4.42824	126.67
6.00600	5.01543	126.64
5.58039	4.65960	126.62
28.66004	23.94259	126.73
2.83859	3.20560	126.71
5.85449	4.88930	126.69
25.65029	21.42146	126.70

Mean 126.68
If $O=16$, Te =127.63

These last values are essentially identical with those found by Pellini. The memoir contains criticisms of the recent work of others upon the atomic weight of tellurium, and is unusually complete.

Tungsten.

Taylor (Doctoral Thesis, University of Pennsylvania, 1901), working under the direction of Edgar F. Smith, has made numerous experiments upon the reduction of WO_3 to W , and the reverse oxidation, and has obtained discordant data as to the atomic weight of tungsten. His material was prepared from wolfram, and purified by recrystallisation as ammonium tungstate. The discrepancies are probably to be explained by the presence in the latter salt of small quantities of an ammonium-iron-manganese tungstate. Experiments were also carried out upon the expulsion of CO_2 from Na_2CO_3 during solution in the latter of WO_3 . This work was done somewhat imperfectly, and the results are not of absolute value; they are, however, encouraging, and show that the method has some presumptive merit. The thesis is essentially a study of methods, rather than of actual determinations of atomic weight. It has value, as showing some of the errors which have vitiated former work, and which may now be avoided.

Uranium.

Aloy (*Comptes Rendus*, cxxxii., 551; also, more fully, in *Ann. Chim. Phys.*, [7], xxiv., 418) has determined the

* *Journal of the American Chemical Society*, vol. xxiv., No. 3.

atomic weight of uranium by a new method, which he proposes to apply to other metals as well. The pure nitrate, of unknown weight, was the material studied. Nitrogen was determined volumetrically, and in the same sample the metal was estimated as UO_2 . From the ratio between these measurements the desired atomic weight was computed. The data given are as follows:—

No.	Volume of N. C.c.	Atomic weight U.
1	15'25	239'3
2	33'5	239'4
3	38'0	239'6
4	52'5	239'5
5	81'25	239'4
6	125'0	239'5
7	151'2	239'4
8	165'0	239'4

The value finally adopted was $U = 239'4$ when $N = 14'04$. The weights of UO_2 obtained are not published in the paper, so that the accuracy of the work cannot well be estimated. Data of this kind, which cannot be recomputed by the reader, are of very little value. It is to be hoped that the paper is only a preliminary statement, and that the full details of the research will appear later.

Lanthanum.

Brauner and Pavlicek (*Proc. Chem. Soc.*, xvii., 63), in determining the atomic weight of lanthanum by synthesis of the sulphate from the oxide, find that the sulphate is always contaminated by Wyrouboff's acid sulphate to a notable extent. The latter salt is so stable that even at 500° some of it remains undecomposed, while another portion of sulphate in the same crucible may have been reduced to a basic compound. Hence all "equivalent" determinations of rare earths hitherto made by the sulphate method are vitiated by this error. Applying, by actual measurement, the necessary correction for excess of acid, and also correcting for the absorption of hygroscopic moisture by lanthanum sulphate, the authors find for the atomic weight of the metal in the most positive fraction of their material, the value $La = 139'0$. Ordinary lanthanum, however, the authors regard as a complex of this true lanthanum with another earth metal.

Praseodymium.

Brauner (*Proc. Chem. Soc.*, xvii., 65) determines the atomic weight of this metal by four methods. First, by analysis of the anhydrous sulphate, whence $Pr = 140'95$. Second, by the analysis of the oxalate, whence $Pr = 140'95$. Third, by synthesis of the sulphate from the oxide, which gave $Pr = 140'78$. Fourth, a series like the third, but with corrections for the presence of acid sulphate and hygroscopic moisture, gave $Pr = 140'93$. The mean result adopted is $Pr = 140'94$. Detailed weighings are not given. An ebullioscopic determination of the molecular weight of praseodymium chloride confirmed the foregoing value.

Neodymium.

Also determined by Brauner (*Proc. Chem. Soc.*, xvii., 66) by means of the sulphate method. Result, corrected for the presence of acid sulphate, $Nd = 143'80$.

Thorium.

By the hydrolysis of thorium oxalate, Brauner (*Proc. Chem. Soc.*, xvii., 68) claims to have decomposed commercial thorium into two earths which he calls Th_α and Th_β . For thorium alpha the atomic weight determinations by the oxalate method gave 233'5, and by the sulphate method 233'3 to 233'7. The most negative fractions of his material, the thorium beta, first gave the value 232'5, which, by further purification was reduced to 232 and 231'9. By continued fractionation an oxide was obtained, giving an atomic weight of $\text{Th}_\beta = 220$. This decrease was accompanied by a reduction in the density of the oxide from 10'2 to 9'6. The complex nature of the

thorium heretofore known may therefore be regarded as proved.

Similar results to those announced by Brauner were independently and almost simultaneously obtained by Baskerville (*Yourn. Am. Chem. Soc.*, xxi., 761). Oxides were prepared, ranging in specific gravity from 8'47 to 11'26, the most definite or real thorium oxide giving values from 9'188 to 9'253. The lowest figures represent an oxide which occurs in small amount in ordinary thorium, the higher to a new earth. The purest thorium compound prepared was a tetrachloride, upon which atomic weight determinations were made, ThO_2 and chlorine being both estimated. Two determinations gave $\text{Th} = 223'2$ and 223'3, or about ten units below the accepted figures. In another sample the value 222'13 was found. To the unknown contamination, which raises the atomic weight of the metal and increases the density of the oxide, an atomic weight of from 260 to 280 must be attributed. This element Baskerville proposes to name carolinium. His paper is confessedly a preliminary communication, and further investigation is in progress. Since his methods of research differ from those of Brauner, the convergence of the results is all the more suggestive.

Older Data.

In Bolton's "Index to Academic Dissertations," published by the Smithsonian Institution, I find the titles of four Russian theses which seem to have escaped the attention of all other writers on atomic weights. Of their value I can say nothing, but as they form part of the literature of the subject I reproduce the titles (as translated in Bolton's work) here:—

Dobrovolsky, V.—"Contributions to the Chemistry of Boron and its Compounds. 1. The Atomic Weight of Boron." Kiev, 1869.

Einbrodt, Paul.—"On the Atomic Weight of Nitrogen." Kharkov, 1840.

Struve, H.—"Dissertation on the Determination of the Atomic Weight of some Elements." St. Petersburg, 1850.

Viluef, V.—"Dissertation on the atomic Weight of Bismuth." St. Petersburg, 1849.

It is to be hoped that some scholar having access to Russian literature may prepare abstracts of these documents, and thereby make the data which are contained in them available for general use.

The Standard of Atomic Weights.

Over this question, controversy still continues. The International Committee of the German Chemical Society publishes its table for 1901 according to both standards; first with $O = 16$, and then in response to a demand from teachers giving a "didactic table" based upon $H = 1$. Both tables are given in this report, at the end. In their third general report (*Berichte*, xxxiv., 4358), the same Committee publish many letters from individual chemists, the final outcome from all sources being 106 voices in favour of $H = 1$ and 78 for $O = 16$. This preponderance is offset by the fact that five societies expressed their views as societies, four for $O = 16$, and only one for the hydrogen basis. The Committee, therefore, continues to recommend the oxygen standard. The members of the Verein deutscher Chemiker who protested against the oxygen standard, also publish the results of their canvass among the teachers of chemistry in German-speaking countries (*Zeit. Angew. Chem.*, February 19, 1901), and give the names of the respondents. 104 favoured the hydrogen unit; 19 preferred the oxygen standard. Erdman (*Zeit. Angew. Chem.*, August 20, 1901) has printed a table of atomic weights with $H = 1$, which is in most points identical with that of your Committee, only it is carried out uniformly to two decimal places. The two values in which it varies essentially from ours is in $\text{Co} = 58'80$, or about $\frac{1}{2}$ unit higher, and in $\text{Pd} = 106$ or $\frac{1}{2}$ lower.

In favour of the oxygen standard there is a paper by

Richards (*Zeit. Anorg. Chem.*, xxviii., 355), and the incidental remarks by Sakurai in his essay upon "Some Points in Chemical Education" (read before the Chemical Section of the British Association, 1901, separately printed; see also CHEMICAL NEWS, vol. lxxiv., p. 151). There is also, on the hydrogen side of the discussion, an elaborate article by Glücksmann (*Zeit. des Allgem. Oesterr. Apotheker-Vereins*, 1901, Nos. 23, 24, 25, 26).

Still another standard of values has been proposed by Hinrichs ("The Absolute Atomic Weights of the Chemical Elements, &c.," published at St. Louis, Mo., 1901), who takes as the experimental basis of his system the atomic weight of "carbon-diamond" as 12 exactly. He seeks to show that all true atomic weights are multiples of the half-unit of hydrogen, but his method of discussion is more polemical than scientific. He reiterates his well-known objections to the work of Stas and the "Stasians," and denounces the conclusions of these men as "false and fraudulent."

Miscellaneous Notes.

Benoist (*Comptes Rendus*, cxxxii., 772), studying the "specific opacity" of substances with regard to the X-rays, finds that for the elements this property is a definite function of the atomic weight. Applying this principle to certain compounds of indium, he shows that the atomic weight of that element must be 113.4 rather than 75.6; that is, the classification of indium as trivalent and not as a dyad is confirmed.

Chabrie and Rengade (*Comptes Rendus*, cxxxii., 472) reach the same conclusion from an ebullioscopic determination of the molecular weight of indium acetyl-acetate.

In the report of this Committee for 1900, Demarcay's work on the atomic weight of samarium was cited. He then found the oxide hitherto studied to have been contaminated with another earth of higher atomic weight. Continuing the investigation, he has isolated this earth (*Comptes Rendus*, cxxxii., 1484), which corresponds to a new metal of atomic weight 151. To this metal he gives the name "Europium."

G. and E. Urbain (*Comptes Rendus*, cxxxii., 136), by a prolonged fractionation of the yttria groups, have obtained yttria and ytterbia in a high degree of purity. The atomic weights of the two metals are given, without details, as—

$$Yt = 88.6.$$

$$Yb = 172.6.$$

These values are presumably based upon O = 16.

A new earth, resembling and associated with zirconia, has been separated by Hofmann and Prandtl (*Berichte*, xxiv., 1064) from euxenite. They assume that the metal is a tetrad, and determine its atomic weight from two analyses of separate fractions of the sulphate.

Weight of sulphate.	Weight of oxide.	Atomic weight.
0.2176	0.1234	177.6
0.1170	0.0664	177.9

These data are only preliminary, and further investigation is promised.

Strutt (*Phil. Mag.*, [6], i., 311) has applied the calculus of probabilities to the tendency exhibited by atomic weights to approximate whole numbers. No atomic weight can vary from a whole number by more than 0.5. Taking the eight elements Br, C, Cl, H, N, K, Na, and S, and assuming O = 16, the sum of the eight deviations from whole numbers is only 0.809. The probability that this sum should not be larger is represented by about one chance in one thousand. Hence the atomic weights tend to approximate the whole numbers to a greater extent than can be accounted for by accident. As Strutt puts the case—"We have stronger reasons for believing in the truth of some modification of Prout's law than in that of many historical events which are universally accepted as unquestionable." A similar discussion, applied to eighteen elements, was once put forth by Mallet, but the mathematical treatment was not the same. There is also a

short paper on the same subject by Rudolphi (*Chemiker Zeitung*, xxv., 1133).

Table of Atomic Weights.

The following table of atomic weights is somewhat differently arranged from that of a year ago. First, your Committee gives its own list, followed by that of the German Chemical Society on the basis of H = 1. Then come the three columns, Clarke, Richards, and Brauner's on thorium, and Brauner's upon the other rare earth metals should be taken into account. All of this work, however, needs to be carried further and published more fully before the table should be correspondingly changed. The magnitude of the necessary changes is as yet too uncertain. In the case of thorium, the atomic weight given relates to that mixture of earths which has hitherto been called thoria, and which we actually meet in analytical operations.

In the light of the foregoing report, some of the values given in the table must be regarded as doubtful. Demarcay's work on samarium, Baskerville's and Brauner's on thorium, and Brauner's upon the other rare earth metals should be taken into account. All of this work, however, needs to be carried further and published more fully before the table should be correspondingly changed. The magnitude of the necessary changes is as yet too uncertain. In the case of thorium, the atomic weight given relates to that mixture of earths which has hitherto been called thoria, and which we actually meet in analytical operations.

	H = 1.		O = 16.		
	Clarke.	German.	Clarke.	Richards.	German.
Aluminum ..	26.9	26.9	27.1	27.1	27.1
Antimony ..	119.5	119.1	120.4	120.0	120.0
Argon ..	39.6	39.6	39.96	39.92	39.9
Arsenic ..	74.45	74.4	75.0	75.0	75.0
Barium ..	136.4	136.4	137.40	137.43	137.4
Bismuth ..	206.5	206.9	208.1	208.0	208.5
Boron ..	10.9	10.9	11.0	11.0	11.0
Bromine ..	79.35	79.36	79.95	79.955	79.96
Cadmium ..	111.55	111.6	112.4	112.3	112.4
Cæsium ..	131.9	132.0	132.9	132.9	133.0
Calcium ..	39.8	39.7	40.1	40.1	40.0
Carbon ..	11.9	11.91	12.0	12.001	12.00
Cerium ..	138.0	139.0	139.0	140.0	140.0
Chlorine ..	35.18	35.18	35.45	35.455	35.45
Chromium ..	51.7	51.7	52.1	52.14	52.1
Cobalt ..	58.55	58.56	59.00	59.00	59.0
Columbium ..	93.0	93.3	93.7	94.0	94.0
Copper ..	63.1	63.1	63.60	63.60	63.6
Erbium ..	164.7	164.8	166.0	166.0	166.0
Fluorine ..	18.9	18.9	19.05	19.05	19.0
Gadolinium ..	155.2	155.0	156.4	156.0	156.0
Gallium ..	69.5	69.5	70.0	70.0	70.0
Germanium ..	71.9	71.5	72.5	72.5	72.0
Glucinum ..	9.0	9.03	9.1	9.1	9.1
Gold ..	195.7	195.7	197.2	197.3	197.2
Helium ..	3.93	4.0	3.96	3.96	4.0
Hydrogen ..	1.000	1.00	1.008	1.0075	1.01
Indium ..	113.1	113.1	114.0	114.0	114.0
Iodine ..	125.89	125.90	126.85	126.85	126.85
Iridium ..	191.7	191.5	193.1	193.0	193.0
Iron ..	55.5	55.6	55.9	55.9	56.0
Krypton ..	81.15	81.2	81.76	81.7	81.8
Lanthanum ..	137.6	137.7	138.6	138.5	138.0
Lead ..	205.36	205.35	206.92	206.92	206.9
Lithium ..	6.97	6.98	7.03	7.03	7.03
Magnesium ..	24.1	24.18	24.3	24.36	24.36
Manganese ..	54.6	54.6	55.0	55.02	55.0
Mercury ..	198.50	198.8	200.0	200.0	200.3
Molybdenum ..	95.3	95.3	96.0	96.0	96.0
Neodymium ..	142.5	142.5	143.6	143.6	143.6
Neon ..	19.8	19.9	19.94	19.94	20.0
Nickel ..	58.25	58.3	58.70	58.70	58.7
Nitrogen ..	13.93	13.93	14.04	14.04	14.04
Osmium ..	189.6	189.6	191.0	190.8	191.0
Oxygen ..	15.88	15.88	16.000	16.000	16.00
Palladium ..	106.2	105.2	107.0	106.5	106.0
Phosphorus ..	30.75	30.77	31.0	31.0	31.0
Platinum ..	193.4	193.3	194.9	195.2	194.8
Potassium ..	38.82	38.86	39.11	39.14	39.15

	H = 1.		O = 16.	
	Clarke.	German.	Clarke.	Richards, German.
Praseodymium.	139'4	139'4	140'5	140'5
Rhodium ..	102'2	102'2	103'0	103'0
Rubidium ..	84'75	84'76	85'4	85'44
Ruthenium ..	100'9	100'9	101'7	101'7
Samarium ..	149'2?	148'9	150'3?	150'
Scandium ..	43'8	43'8	44'1	44'
Selenium ..	78'6	78'5	79'2	79'1
Silicon ..	28'2	28'2	28'4	28'4
Silver ..	107'11	107'12	107'92	107'93
Sodium ..	22'88	22'88	23'05	23'05
Strontium ..	86'95	86'94	87'60	87'6
Sulphur ..	31'83	31'83	32'07	32'065
Tantalum ..	181'5	181'6	182'8	183'
Tellurium ..	126'1	126'	127'7	127'5?
Terbium ..	158'8	—	160'	160'
Thallium ..	202'61	202'6	204'15	204'15
Thorium ..	230'8?	230'8	232'6?	233'
Thulium ..	169'4	170'	170'7	171'?
Tin ..	118'1	117'6	119'0	119'0
Titanium ..	47'8	47'7	48'15	48'17
Tungsten ..	182'6	182'6	184'	184'
Uranium ..	237'8	237'7	239'6	238'5
Vanadium ..	51'0	50'8	51'4	51'4
Xenon ..	127'	127'	128'0	128'
Ytterbium ..	171'9	172'	173'2	173'
Yttrium ..	88'3	88'3	89'0	89'0
Zinc ..	64'9	64'9	65'4	65'40
Zirconium ..	89'7	90'0	90'4	90'6

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING JUNE 30TH, 1902.

By SIR WILLIAM CROOKES, F.R.S.,

and

PROFESSOR DEWAR, F.R.S.

To CHARLES FERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolitan Water Act, 1871.

London, July 8th, 1902.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 192 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from June 1st to June 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 192 samples examined by us chemically during the month, all were found to be clear, bright, and well filtered.

Although there has been a considerable excess of rain in London during the past month, viz., 1'06 inches or 86'0 per cent, the same cannot be said for Oxford, which place we have always taken as being about the centre of the Thames watershed; it must also be remembered that no rain that falls below Hampton, where the intakes are, affects the London water supply derived from the Thames. The actual rainfall recorded at Oxford during June was

only 1'96 inches, the average fall is 2'10 inches, making a deficit of 0'14 inch, which, added to the previous one, makes a total deficit for the first six months of the year of 3'30 inches, or 30'5 per cent on the thirty-five years' average.

Our bacteriological examinations of 509 samples have given the results recorded in the following table; we have also examined 41 other samples, from special stand-pipes, wells, &c., making a total of 550 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 25 samples) ..	312
New River, filtered (mean of 129 samples) ..	16
Thames, unfiltered (mean of 25 samples) ..	4338
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 272 samples) ..	32
Ditto ditto highest	538
Ditto ditto lowest	0
River Lea, unfiltered (mean of 25 samples) ..	464
River Lea, from the East London Company's clear-water wells (mean of 33 samples) ..	35

Of the 434 samples taken daily from the filter-wells of the Metropolitan Water Companies, and examined bacteriologically by us, fifteen samples, or 3'4 per cent, were sterile. Thirteen samples contained more than 150 microbes, while twenty-five samples, or 5'7 per cent, contained more than 100 microbes per c.c. The mean number of microbes in the twenty-five excess samples was 206, against a corresponding mean of 173 in thirty excess samples during May. This increase was caused by the sudden rainfall at the beginning of the month, when the microbic impurity in the raw Thames Water rose very considerably, thereby throwing extra strain on the filters. Special bacteriological examinations have revealed the presence of none but harmless river microbes.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.

JAMES DEWAR.

ACTION OF BROMINE ON THALLOUS CHLORIDE IN THE PRESENCE OF ORGANIC SOLVENTS, AND BY THE DRY METHOD.

By V. THOMAS.

As has been pointed out in previous papers, we cannot easily obtain the intermediate chlorobromides, regularly formed by the addition of bromine to thallous chloride, when working in contact with water. As we have said, the cause of this want of success can be found in the dissolving action of water on the intermediate compounds at first formed.

In the hope of succeeding in the preparation of the chlorobromides, we decided to use as solvents organic compounds such as chloroform, sulphide of carbon, and tetrachloride of carbon. In the presence of these solvents the bromination is immediately shown to be different from the bromination in the presence of water, inasmuch as the fixation of the bromine stops when we reach the type Tl_2X_4 .

A point not less interesting to note is the non-formation of normal compounds of addition. As, when we operate in the presence of water, chlorobromides are always formed less rich in chlorine than the product with which we start; that is to say, containing less than one atom of chlorine for one atom of thallium. It is probable that here again we may admit the transitory formation of normal compounds of addition, of thallosolthallous chlorobromide, for example.

These in the presence of solvents do not undergo any dissociation, as when in contact with water, into thallic compounds or compounds containing less of the halogen,

but they enter into reaction with the solvent itself in a more or less complex manner.

I. *Action of Bromine on TiCl in the presence of Chloroform.*—4.8 grms. of thallous chloride, 2.8 grms. of bromine, and 40 grms. of the solvent were left for a long time in a ground stoppered flask. The thallous chloride takes a yellow tint, which at first increases in intensity and then seems to become slightly paler.

Analysis.—Found, AgCl+AgBr, 104.31, and 104.02; Cl, 6.76 and 7.80; Br, 30.67 and 30.98. Calculated for $Tl_2Cl_2Br_2$:—AgCl+AgBr, 103.58; Cl, 7.08; Br, 31.90.

As will be seen, the analysis shows a slight excess of chlorine; the cause for this will be found in the difficulty experienced in effecting the transformation in a complete manner.

If we evaporate the solution from which the yellow powder has been separated, we obtain fine yellow needles of the same composition.

II. *Action of Bromine on TiCl in the presence of Sulphide of Carbon.*—Sulphide of carbon is a very bad solvent in which to effect the bromination of thallous chloride. The reaction is very complex.

We might suppose that two phenomena are produced at the same time; on the one hand, the bromination of thallous chloride and the setting free of the chlorine, and on the other hand, the action of the halogens on the sulphide of carbon.

The part played by the solvent can be demonstrated in a very simple manner; it is sufficient to leave a small quantity of chloride of thallium, sulphide of carbon, and bromine in a test-tube for several days. Fumes having the very characteristic odours of halogen compounds of sulphur are given off. After a sufficient time, especially if we use a sufficient quantity of bromine, the tube commences to show long needles. These needles do not contain thallium, but apparently represent compounds analogous to those mentioned by Hell and Urech (*Ber.*, vol. xv., p. 909; and vol. xvi., p. 1144), for which these chemists proposed the formulæ CS_2Br_4 , $C_2Br_6S_3$, $C_9Br_4S_4+2H_2O$.

It is certain that in such a reaction the thallous chloride is principally a "carrier" for the halogen; that is an action that has long been well known. In trying to discover what becomes of the thallous salt originally added in this reaction, we readily observe that it is not confined to assisting the halogenation of the sulphide of carbon. It itself comes into play, and is found in the products of the reaction in the form of the dibromide, $TlBr_2$, more or less impure.

By adopting suitable conditions we can obtain a yellow product containing:—Found, Cl, 0.23; Br, 43.31. Calculated, Cl, 0.00; Br, 43.9.

In this reaction the thallous chloride is susceptible of completely exchanging its chlorine for bromine. Thus it resembles ferric chloride, which, as we have shown, is able, even in the aromatic series, to effect the substitution of the chlorine for bromine or iodine (*Comptes Rendus*, July 18, 1898).

III. *Action of Bromine on TiCl in the presence of Tetrachloride of Carbon.*—Here the same phenomena are observed as when employing chloroform for the solvent. The analyses all show a considerable excess of bromine and a deficit of chlorine. Found, Br, 33.97 and 33.50; Cl, 6.13 and 6.33. Calculated for $Tl_2Cl_2Br_4$:—Br, 31.90; Cl, 7.08.

Possibly in effecting the bromination under special conditions, with heat for instance, we might succeed in replacing the whole of the chlorine by bromine.

However this may be, if we use an impure tetrachloride containing sulphide of carbon even in very small quantities, we immediately notice the presence of the halo-derivative of sulphur; at the same time almost complete bromination takes place. In this manner we have obtained a product corresponding to the following composition:—Found, Br, 43.10; Cl, 0.28. Calculated for $TlBr_2$:—Br, 43.90; Cl 0.00.

IV. *Action of Bromine on Thallous Chloride by the Dry Method.*—When, instead of operating in the presence of a solvent, we use the dry method, it is easy to show that:—(1) Normal compounds of addition are formed; (2) the ultimate term of bromination corresponds, under ordinary conditions, to the type $TlClBr$.

This chlorobromide is the one constantly obtained either with bromine and the chloride in the cold, or at a higher temperature, in sealed tubes for instance.

The only really practicable method for preparing this body consists in heating the materials in sealed tubes at 100–125°. In this manner we obtain a product which on analysis gives the following figures:—Found, Br, 25.50 and 24.86; Cl, 11.06 and 10.61. Calculated for $TlClBr$:—Br, 25.03; Cl, 11.11.

This compound shares the properties of both the corresponding chloride and bromide. It is of a deeper yellow than the chloride. Like this salt, its colour rapidly becomes darker on heating and regains its original tint on cooling. Water decomposes it rapidly with the formation of a chlorobromide of the type Tl_2X_6 .—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 11.

EXPERIMENTAL RESEARCHES ON THE CONSTITUTION OF CRYSTALS.*

By A. E. TUTTON, B.Sc., F.R.S., F.C.S.

(Continued from p. 32).

In turning now to the consideration of the optical characters of the crystals of the series of salts in question, it will be necessary to remember two main facts. The first is, that the symmetry of the rhombic system, in which the sulphates and selenates of the alkalis crystallise, determines that the velocity of light transmission shall be different along the three morphological axes of the crystals, and that two of these directions shall be those of maximum and minimum velocity respectively. In other words, the ellipsoid which, as is well known, in general represents the velocity of light transmitted through a crystalline medium, is one whose three rectangular axes are of unequal lengths, but which is fixed in direction, for these axes are identical in direction with the three morphological axes. The second is, that in the case of the monoclinic double sulphates and selenates the symmetry of the system demands that only one of the three rectangular axes of the optical ellipsoid shall be identical with a morphological axis, the latter being the one symmetry axis of the system. The ellipsoid may therefore be regarded as free to rotate about this axis, for the inclination of the other two rectangular axes of the ellipsoid, which lie in the plane of symmetry, may be any whatsoever with respect to the two inclined morphological axes which lie in that plane.

Within the three models of sections of our rhombic sulphate crystals you now see three illuminated ellipses. They represent the sections of the three optical ellipsoids; but in this case the inner one corresponds to the cesium salt, and represents the ellipsoidal line to which light waves emanating from the imaginary centre of the crystal would penetrate in a given interval of time. The middle one shows the distance to which they would penetrate if the crystal were one of rubidium sulphate or selenate; and any point on the outer one, which is much nearer to the middle one than the outer one is, represents the position at which the light waves would arrive in the same interval of time if the crystal were one of potassium sulphate or selenate.

The velocity with which light travels through the crystals of the three salts is thus observed to vary in the same manner as does the atomic weight of the alkali metal present in the salt.

* A Lecture delivered at the Royal Institution of Great Britain, Friday, May 2, 1902.

In the case of the monoclinic double sulphates and selenates, we have a further phenomenon of even greater interest exhibited. For not only does the velocity along the three rectangular axes of the ellipsoid vary with the atomic weight, but the possible rotation of the whole ellipsoid about the symmetry axis is found to actually occur; and to occur, moreover, to a very considerable extent, sometimes amounting to as much as 20° .

Further, most interesting of all, the rotation varies in a perfectly regular manner throughout all the triplets, in accordance with the atomic weight of the alkali metal present in the salt. This may be demonstrated to you by means of the lantern slide which you now see projected on the screen. You observe the outline of a crystal, arranged so that the screen represents the symmetry plane, and within it an ellipsoid. The latter is at present arranged as it is usually situated in any potassium salt of the series, its major axis being inclined so that its top is somewhat to the left of the vertical morphological axis. The ellipsoid will now be rotated to the approximate position, further on the left of the vertical axis, which it occupies in any rubidium salt of the series, by means of a simple mechanical device; and again it is rotated much further, to about the situation which it takes up in any caesium salt.

In this phenomenon the accelerating progression according to the atomic weight of the alkali metal is beautifully exhibited.

It may now interest you to learn how these results have been obtained. We have to determine, first the position of the optical ellipsoid, and next its dimensions. That is to say, we have to determine the velocity of light transmission in all the various directions in the crystal. Determinations of the refractive index in different directions will afford us the necessary data, and if we can discover the directions of maximum and minimum velocity we shall obtain all that we require if we make the determinations of refractive index for these two directions, and for a third direction at right angles to the plane containing them. For these three directions will be those of the three axes of the ellipsoid.

It will be next demonstrated to you how we discover the positions of the axes of the optical ellipsoid.

It is scarcely necessary to introduce to a Royal Institution audience the magnificent pair of Nicol prisms which formerly belonged to the late Mr. Spottiswoode. The analysing Nicol is at present arranged with its vibrating direction parallel to that of the polarising prism, and to the horizontal cross-wire which you see appearing on the screen, so that light passes to the screen.

There is also focussed the outline of a section of a monoclinic crystal, placed between the two Nicols, and which you may take as representing a crystal of one of our double sulphates or selenates; for it behaves precisely similarly, and is immensely larger than any crystal that could be obtained of one of those salts. We now rotate the analyser so that its vibrating direction is at right angles to that of the polariser, when you see the crystal section brilliantly coloured on the dark field.

If, however, we rotate the crystal section in its own plane, you observe that the coloured light becomes weaker, until, at a certain position, it is altogether extinguished. Further rotation causes light to again appear, and if we complete the circle of rotation we shall find that the crystal becomes four times dark and four times light. That is, there are two directions, at right angles to each other, in which extinction occurs. These two directions are those of two axes of the optical ellipsoid. If the crystal is so prepared as to show on its edge traces of one or two natural faces, and if our rotating stage is divided, it is quite easy to determine the angle between any given trace of a face and either of the extinction directions. In the case of monoclinic crystals the section is cut parallel to the symmetry plane, and the reference edge will usually be a trace of one of the faces in the primary zone perpendicular to the symmetry plane. That

is the case with the two faces whose traces you see are left after the grinding of the large section you observe on the screen. A much more refined method of measuring the angle between the extinction direction and the basal plane based on this principle, was employed in the research.

We will next illustrate the determination of the three refractive indices, corresponding to the three velocities along the three axes of the ellipsoid. There is here a large 60° -prism of a crystal which behaves similarly to the salts we are discussing, and which has been specially cut and polished for this lecture by Mr. Hilger. The refracting edge is parallel to an axis of the ellipsoid. Employing it in the usual manner to throw a spectrum on the screen, we observe that instead of a single spectrum, as when we use a prism of glass, we have two spectra produced, differing considerably in their dispersion. Moreover, on placing a Nicol prism in the path of the light, we see that these spectra consist of polarised light, for one extinguishes when the Nicol is arranged with its vibration direction at 0° , and the other when the Nicol is at 90° . One of the spectra, in fact, is formed by light vibrating parallel to the refracting edge, and therefore to that axis of the ellipsoid which runs parallel with it, and the other by light vibrating at right angles to that direction. If, in cutting our prism, we make this perpendicular direction to coincide with a second axis of the ellipsoid, the two spectra, when set to their minimum deviation, will, provided we know also the angle of the prism, at once afford us the data for computing the refractive indices corresponding to the two axes of the ellipsoid in question. A second prism, similarly cut so as to have vibration directions parallel to one of these axes and to the third axis, will afford us the third refractive index as well as a repetition of one of the first two.

You observed that the two spectra are considerably separated on the screen. If the two axial directions are those of maximum and minimum velocity, the amount of separation is a measure of the double refraction. Now it has been observed that the amount of the double refraction also varies progressively according to the atomic weight, and this fact may be illustrated realistically by means of a carefully made lantern-slide, reproducing the actual positions, as seen in the spectrometer, of the two spectra afforded by a triplet of salts. The triplet chosen consists of potassium zinc sulphate, rubidium zinc sulphate, and caesium zinc sulphate. The spectra are represented by the images of the spectrometer slit for two wave-lengths of light, those corresponding to the red and greenish-blue hydrogen lines. The spectra were produced by three prisms of the respective salts, of precisely the same angle, near 60° . You see that the two upper spectra, representing the potassium salt, are the furthest apart, and that the separation diminishes for the rubidium salt, and it becomes so much less in the case of the caesium salt that the two lower spectra formed by this salt partially overlap.

This relative behaviour as regards double refraction is quite general throughout all the four series of salts.

The point can be further illustrated by the curves which you now see projected on the screen. The two outer curves represent the maximum and minimum refraction, and the inner one the intermediate refraction along the third rectangular axis of the ellipsoid. You observe that the two outer curves converge towards each other as the atomic weight of the alkali metal increases.

This leads in four particular cases to perhaps the most interesting of all the results derived from the work. We will illustrate it by the case of caesium magnesium selenate. The curves which you now see on the screen are those for the magnesium triplet of double selenates. On arrival at the caesium salt the convergence has actually brought the curve for the minimum refraction into contact with that for the intermediate refraction, so that we have here a crystal whose total double refraction is exceptionally small, and in which the velocities along

two of the three ellipsoidal axes are approximately equal, which is characteristic of tetragonal and hexagonal crystals, but a perfect anomaly in a monoclinic crystal. Let us see, however, if the identity is a fact for all wave-lengths, as would be the case in a truly uniaxial crystal of tetragonal or hexagonal symmetry. We will reproduce for you the appearance in the spectrometer afforded by a prism so cut as to give us the two spectra corresponding to these two intersecting curves; that is, whose vibration directions are those of the two apparently equal axes of the velocity ellipsoid.

The images in three colours which you see are those corresponding to the wave-lengths of red lithium light, green thallium light, and violet hydrogen light. You observe that they completely overlap, and if we only used the same magnification as we used for the other images we saw just now, you would say that they are absolutely identical images. We are attempting to faithfully reproduce the whole phenomena by the use of separate slides, shown by two equal lanterns, and when one of them is shut off, the effect is exactly as when a Nicol is introduced at 0° in the spectrometer, which extinguishes one of the spectra. When the other is shut off instead, what you observe is the same as if we rotated the Nicol to 90° , which would extinguish the second spectrum. Commencing with both lanterns on, as when no Nicol is being used, or if it is, it is arranged at 45° , and examining the images closely, we notice that the red and violet images are distinctly double, while the central green image is a truly single one. Now we cut off one lantern, and you please imagine that we are introducing a Nicol at 0° ; the effect has been that the outermost image in the case of both red and violet has disappeared. Now shutting off the other lantern instead, and you please imagine we are rotating the Nicol to 90° , the innermost images disappear. All this time the green image remains fixed and single. This evidently means that for the middle part of the spectrum there is absolute identity of refraction and therefore of velocity, while for the red end there is a minute difference of refraction in one sense, and for the violet end a similar small difference in the opposite sense. The fact is, the two images, corresponding to the two ellipsoidal axes, for red are slightly separated; they approach and coalesce for green; then they pass each other and re-separate on the opposite side of each other as violet is approached.

Thus our crystal is only truly uniaxial for one wave-length of light, and this apparent anomalous refraction is merely the effect of the operation of the rule of progression.

You will have gathered that for the prosecution of this work it has been necessary to prepare some hundreds of 60° -prisms and parallel-sided section-plates, all accurately cut to the desired orientation with respect to the crystal faces. For this purpose it has been found necessary to have the delicate apparatus constructed which you see before you on the table, and a photograph of which is also thrown on the screen. The crystal held in a grip-holder, is suspended from a refined apparatus which serves not only for the adjustment of a zone of the crystal's faces to the vertical axis of the instrument, as determined by the observer through the telescope of the collimator signal-lit, but also, as the movements are graduated, for its setting to any position with respect to the axis. Separate and interchangeable cutting and grinding gear are provided, and also a delicate means of varying the pressure of the crystal on the grinding disc, so that the most fragile crystals can be manipulated without danger of fracturing them.

It is not too much to say of this instrument, that without it the work described to you this evening would never have been possible.

Another original instrument which you see before you is an apparatus for producing spectrum monochromatic light of any wave-length whatsoever. For all the optical researches have to be carried out in pure monochromatic light; that is, for a series of colours of light, each of

which is composed of vibrations of as nearly one wave-length as possible; for all the optical constants vary considerably for different wave-lengths of light. It is essentially a spectroscopy constructed to transmit as large a proportion of the light as possible which streams from the condenser of an electric lantern; a broad spectrum is produced by a highly refractive prism, and is focussed on the back of a second slit, which permits only a selected line of the spectrum to escape, in the same manner as in the well-known apparatus of Sir Wm. Abney. By rotation of the prism the spectrum is moved over the exit slit, so as to permit any desired colour to escape, whose wave-length is known from the reading of the calibrated circle on which the prism is mounted.

The apparatus is placed before you just as it is arranged, in front of the goniometer-spectrometer, when determining refractive indices for a series of different wave-lengths.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, Wednesday, June 18th, 1902.

Prof. EMERSON REYNOLDS, Sc.D., V.P.R.S., President, in the Chair.

(Concluded from p. 34).

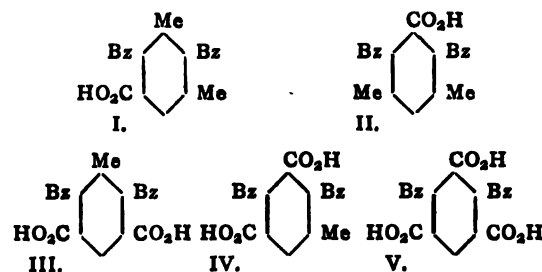
*113. "Derivatives of Dibenzoylmesitylene." By W. H. MILLS and T. H. EASTERFIELD.

The authors described a method of preparing dibenzoylmesitylene by which it may be obtained in quantity, and methods for the production and isolation of the five acids which result by the successive oxidation of its methyl groups, namely, *asym.* dibenzoylmesitylenic acid, m. p. 174° , *sym.* dibenzoylmesitylenic acid, m. p. 222° , *asym.* dibenzoylulvic acid, m. p. 211° , *sym.* dibenzoylulvic acid, m. p. 262° , and dibenzoyltrimesic acid, m. p. 250° .

A mixture of these, of which *asym.* dibenzoylmesitylenic acid is by far the largest constituent, is obtained by boiling the ketone with dilute nitric acid the boiling-point of which has been raised by the addition of large quantities of potassium and sodium nitrates.

The two dibenzoylulvic acids are most conveniently prepared by treating the easily obtainable *asym.* dibenzoylmesitylenic acid with one equivalent of potassium permanganate in alkaline solution. If double this amount of potassium permanganate be used, dibenzoyltrimesic acid is obtained.

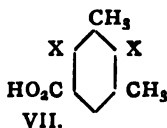
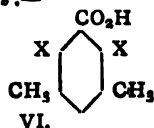
The constitution of these acids readily follows from the following facts:—



The dibenzoylmesitylenic acid of melting-point 174° , oxidised with potassium permanganate, yields a mixture of both dibenzoylulvic acids. It must therefore be the asymmetrical acid of formula I. The dibenzoylmesitylenic acid of melting-point 222° , similarly treated, yields only one dibenzoylulvic acid melting at 211° . Hence, it must

be represented by formula II., and the dibenzoylviolic acid resulting from it by IV. The dibenzoylviolic acid melting at 262° must therefore be represented by III., and for dibenzoyltrimesic acid only one formula is possible, namely, V.

In working with these acids, the fact was discovered (also noticed by Graebe, *Ber.*, 1900, xxxiii., 2026) that diortho-substituted acids do not obey V. Meyer's esterification rule when the ortho-substituents are benzoyl groups:—



whereas it follows from V. Meyer's work that the esterification constant of an acid of formula VI. would in general be indefinitely smaller at the ordinary temperature than that of an acid of formula VII., in the case where X is a benzoyl group it is smaller only in the ratio of approximately 0.65 to 1.0.

114. "The Molecular Condition of Borax in Solution." By H. S. SHELTON.

Borax (Shields, *Phil. Mag.*, 1893, xxxv., 365) is slightly hydrolysed into sodium hydroxide and boracic acid. Experiments on the diminution of conductivity on the addition of further quantities of boracic acid show an increase of this hydrolysis when further diluted with water, and also with increase of temperature. In N/10 solution, the hydrolysis was estimated by Shields to be 0.5 per cent at 25° , and the experiments of the author with N/200 solution showed 4 per cent at 25° , and 6 per cent at 50° . Kahlenberg and Schreiner (*Zeit. Phys. Chem.*, 1896, xx., 547) concluded from freezing-point determinations that six chemical individuals are present in a dilute solution of borax.

These chemical individuals seem to be $2\text{H}_3\text{BO}_3, 2\text{NaBO}_2$, + -

as shown by the following equation:—



The author has confirmed this by a number of direct experiments, amongst which the most important is the precipitation of AgBO_2 , and determination of the boracic acid remaining in the solution.

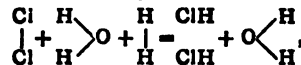
115. "On the Union of Hydrogen and Chlorine." Part V. By J. W. MELLOR, D.Sc.

There is no experimental evidence to show that chlorine gas under the influence of light undergoes any change capable of appreciably affecting the chemical activity of that element towards hydrogen. Part of the energy absorbed by moist chlorine from sunlight is dissipated as heat. This causes the Budde effect. Under the influence of light, chlorine sets up and maintains in a state of equilibrium a reversible reaction with water vapour, possibly $2\text{H}_2\text{O} + 2\text{Cl}_2 \rightleftharpoons 4\text{HCl} + \text{O}_2$. Dry chlorine does not exhibit the Budde effect. The rise in temperature of imperfectly dried chlorine when exposed to sunlight appears to be due to some chemical reaction between the moisture and the chlorine gas. A layer of moist chlorine just thick enough to screen a bulb of mixed hydrogen and chlorine gases from chemical action is not sufficient to prevent chemical action if the chlorine is dried by means of purified phosphorus pentoxide. The actinic energy continuously absorbed from sunlight by moist chlorine is dissipated in at least three ways:—(1) In maintaining the above chemical reaction; (2) by conversion into heat during molecular impacts; (3) as external non-actinic radiations from the molecules moving in their free path between molecular collisions.

116. "On the Union of Hydrogen and Chlorine." Part VI. By J. W. MELLOR, D.Sc.

If the reaction between hydrogen and chlorine in the presence of moisture is assumed to take place with the

formation of an intermediate compound, the period of induction is a direct consequence of the law of mass action. Since neither chlorine monoxide nor hydrogen hypochlorite abbreviate the period of induction, neither of these substances can take part as intermediate compounds in the reaction between hydrogen and chlorine. Since chlorine acquires no appreciable chemical activity by exposure to sunlight, the presence of hydrogen as well as of moisture determines the greater chemical activity of an induced mixture of hydrogen and chlorine gases. If an intermediate compound takes part in the reaction between hydrogen and chlorine in the presence of moisture, the most probable "compound" satisfying the required conditions contains $x\text{Cl}_2, y\text{H}_2\text{O}, z\text{H}_2$, where x, y, z are positive integers. The explanation offered by the two dependent reactions involved in the equation,—



remains to be studied.

117. "On some Hydroxy-pyrone Derivatives." By T. TICKLE and J. N. COLLIE, F.R.S.

During some work on the constitution of meconic acid, the authors found that it was necessary to prepare certain hydroxypyrone derivatives in order to compare their properties (especially their colour reactions with ferric chloride) with those of meconic acid.

Dimethylpyrone, when treated with hydrogen peroxide in presence of a ferrous salt, yields hydroxydimethylpyrone, $\text{C}_7\text{H}_8\text{O}_2 + \text{H}_2\text{O}_2 = \text{C}_7\text{H}_8\text{O}_3 + \text{H}_2\text{O}$. This substance crystallises from water in colourless needles; it can be sublimed without decomposition, and melts at 162.5° . With ferric chloride it gives a deep violet-blue colour. Its monacetyl derivative melts at 98° , and does not colour an aqueous solution of ferric chloride.

Meconic acid, when treated in the same manner with hydrogen peroxide, gives carbon dioxide and hydroxycoumaric acid, $\text{C}_7\text{H}_4\text{O}_7 + \text{H}_2\text{O}_2 = \text{C}_6\text{H}_4\text{O}_6 + \text{CO}_2 + \text{H}_2\text{O}$. Hydroxycoumaric acid seems to be a dibasic acid; it gives an intense violet with ferric chloride, melts at 275° , and crystallises from water in long, fine needles. It seems probable that this is the same acid as that first obtained by Reibstein (*Journ. Pr. Chem.*, 1881, [2], xxiv., 286) by the action of barium hydroxide on bromocoumaric acid. He, however, gives no melting-point for the acid, but only for the ethyl ester, 204° , and for the diacetoxy derivative, 75° . The authors find that these derivatives prepared from this acid melt at 207.5° and 75° respectively.

All attempts to prepare either meconic acid or hydroxycoumaric acid by oxidation of chelidonic acid were unsuccessful.

118. "The Absorption Spectra of Phloroglucinol and some of its Derivatives." By W. N. HARTLEY, D.Sc., F.R.S., J. J. DOBBIE, D.Sc., M.A., and A. LAUDER, B.Sc.

Phloroglucinol reacts with some reagents as a phenol, with others as a ketone. It is impossible, therefore, from its chemical behaviour to decide whether its oxygen atoms are present in hydroxyl groups or in direct union with carbon. The trimethyl derivative of phloroglucinol (m. p. 52°) is regarded as a true ester, since its methoxy groups are split off by heating with hydrogen iodide. The absorption spectra of this substance and of phloroglucinol were found to be almost identical. From this it follows that if the structure of the ester has been correctly established, phloroglucinol also must have the enolic structure (*Trans.*, 1899, lxxvi., 640; 1900, lxxvii., 839). Aqueous and alcoholic solutions of phloroglucinol show both general and selective absorption. A layer 50 m.m. thick of a solution containing 1 milligram.-mol. in 20 c.c. water absorbs all rays beyond λ 3000, whilst a layer 5 m.m. thick absorbs all rays beyond λ 2802. The absorption-band is not strongly marked; it appears first in a layer 3 m.m. thick of the solution containing 1 milligram.-mol. in 100 c.c. water, and nearly dies out in a layer 2 m.m. thick, al-

though its position is still distinctly traceable by the weakness of the spectra in solutions of greater dilution. The resemblance between the spectra of phloroglucinol and the trimethyl derivative is so close that phloroglucinol cannot consist of a mixture of the enolic and ketonic forms.

The specimens of phloroglucinol examined were prepared from five different sources, namely, kino, maclurin, resorcinol, phenol (by fusion with sodium hydroxide), and phloroglucinoltricarboxylic ester. The specimens from these various sources had the same melting-point. The melting-point is greatly influenced by the rate of heating, varying between 210° and 217° . The absorption curves of all the specimens agree perfectly. The authors conclude from the identity of the spectra that the structure of phloroglucinol, no matter from what source it is obtained or by what method it is prepared, is always the same.

The spectra of pyrogallol were also re-examined, and the curve was found to resemble very closely that of phloroglucinol, thus confirming the conclusion that the latter substance has the enolic structure.

119. "Solubility of Mannitol, Picric Acid, and Anthracene." By A. FINDLAY.

Determinations of the solubility of mannitol in water, of picric acid in water and in benzene, and of anthracene in benzene, have been made at several temperatures between 25° and 60° .

It has already been shown by the author (*Proc. Roy. Soc.*, 1902, lxi., 471) that if the solubility curve of one substance is known, that of another substance can be calculated from two, or better, three, determinations of the solubility of the second substance at different temperatures. The formula used for this purpose is one analogous to that employed by Ramsay and Young for the calculation of vapour pressures, and has the form $T_1/T_2' = T_2/T_2' + c(t' - t)$, where T_1/T_2' and T_2/T_2' denote the absolute temperatures at which the two substances have the same solubility, c is a constant, and t' and t are the temperatures at which one of the substances has the solubility corresponding to the absolute temperatures T_1 and T_2 or T_1' and T_2' . This formula has been employed in the present case for calculating the solubilities of the above substances between 0° and 25° , and between 60° and 100° (in the case of anthracene and picric acid, in benzene up to the boiling-point of the latter).

120. "Menthyl Formylphenylacetate." By J. B. COHEN and S. H. C. BRIGGS.

A short time ago, the authors commenced a similar investigation to that of Lapworth and Hann (*Proc. Chem. Soc.*, xviii., 144) with the object of studying tautomeric changes by means of optically active substances, in the course of which they prepared menthyl formylphenylacetate described by Lapworth and Hann. As the authors do not intend, under the circumstances, to pursue this line of inquiry, they desire to record their observations, which, though they confirm on the whole the results given in the paper referred to, show some slight differences. Menthyl formylphenylacetate was prepared as follows:—Sodium was allowed to act on a mixture of menthyl acetate and ethyl formate dissolved in dry ether. In this reaction, an interchange occurs to some extent between the menthyl and ethyl groups, so that some menthyl formate, and some ethyl formylphenylacetate are produced. The product was poured into water, the ethereal layer removed, and the aqueous portion acidified and shaken out with ether. The ether was evaporated in a vacuum, and crystals of menthyl formylphenylacetate separated on standing. The menthyl ester thus obtained was in the form of large, well-formed, tetragonal crystals consisting of four-sided prisms terminated by pyramids; purified by re-crystallisation from chloroform, it melted at $82-84^{\circ}$ (L. and H. give $82-83^{\circ}$). The specific rotation of a freshly prepared chloroform solution containing about 3 grms. of substance in 100 c.c. is $[\alpha]_D^{18} = -72.6^{\circ}$ (L. and H. give -74.6°). In an alcoholic solution containing 2 grms. in 100 c.c., the specific rotation was $[\alpha]_D^{18} = -63.9^{\circ}$ (L. and H. give

-64.9°). The addition of sodium ethylate did not affect the rotation. The following were the effects of an alcoholic solution of ferric chloride on the substance when dissolved in various solvents. A faint violet colouration was produced in methyl and ethyl alcohol, ether, and light petroleum. No colouration was produced at first in benzene, chloroform, or ethyl acetate, but slowly developed. Menthyl phenylacetate, obtained in the preparation of the formylphenylacetate, is a colourless and odourless liquid which boils at 216° (39 m.m.).

*121. "Transformation of Diacetanilide into Aceto-p-amino acetophenone." By F. D. CHATTAWAY.

A large number of derivatives of primary aromatic amines in which various atoms or groups are attached to the nitrogen readily undergo intramolecular transformation and form isomerides in which the atom or group is attached to the nucleus in the para- or ortho-position. The best known examples of such changes are those in which a halogen atom, a nitro-group, a sulphonic group, or a hydroxyl group passes on to the ring. So far, however, the commonest substituting group in amines, the acetyl group, has not been observed to wander. The author described experiments in which this group passed into the ring in the normal manner. The substance studied was diacetanilide, which underwent transformation into aceto-p-aminoacetophenone.

122. "Nitrogen Chlorides and Bromides Derived from Ortho-substituted Anilides." By F. D. CHATTAWAY and J. M. WADMORE.

Nitrogen halogen derivatives of orthochlorobenzanilide and orthobromobenzanilide, which have not hitherto been described, are easily obtained by treating the corresponding anilides with an excess of hypochlorous or hypobromous acid, the actions, as in other similar cases, being reversible. The nitrogen chlorides and bromides so obtained show all the typical nitrogen halogen group reactions, and readily undergo transformation into the isomeric 2:4-disubstituted anilides.

The following compounds have been prepared:—

Benzoyl o-chlorophenyl nitrogen chloride,—



colourless, six-sided plates, m. p. 94° ; Benzoyl-o-chlorophenyl nitrogen bromide, $C_6H_4Cl \cdot NBr \cdot COC_6H_5$, short prisms of a deep yellow colour, m. p. 110° . o-Bromobenzanilide, $C_6H_4Br \cdot NH \cdot COC_6H_5$, colourless rectangular plates, m. p. 116° . Benzoyl-o-bromophenyl nitrogen chloride, $C_6H_4Br \cdot NCl \cdot COC_6H_5$, colourless, transparent prisms, m. p. 85° . Benzoyl-o-bromophenyl nitrogen bromide, $C_6H_4Br \cdot NBr \cdot COC_6H_5$, yellow, six-sided, rhombic prisms, m. p. 99° . Acetyl-o-chlorophenyl nitrogen bromide, $C_6H_4Cl \cdot NBr \cdot COCH_3$, pale yellow, four-sided prisms, m. p. 152° . Acetyl-o-bromophenyl nitrogen chloride, $C_6H_4Br \cdot NCl \cdot COCH_3$, colourless, four-sided prisms, m. p. 86° .

123. "Substituted Nitrogen Chlorides containing the Azo-group." By F. D. CHATTAWAY.

In order to determine the effect of an azo-group on the properties and direction of transformation of nitrogen chlorides, a number of such compounds have been prepared from aminoazobenzene. Aminoazobenzene yields well crystallised acetyl propionyl and benzoyl derivatives, which react with hypochlorous acid very readily, and form nitrogen chlorides possessing all the characteristic properties of the nitrogen halogen group. This adds another to the many reasons for regarding aminoazo-compounds as primary amino-derivatives.

The following compounds have been prepared:—

p-Acetylchloroaminoazobenzene,—



orange-red plates, m. p. 113.5° . Propionyl-p-aminoazobenzene, $C_6H_5 \cdot N:N \cdot C_6H_4 \cdot NH \cdot COC_2H_5$, large, orange-red plates, m. p. 170° . p-Propionylchloroaminoazobenzene,

$C_6H_5 \cdot N:N \cdot C_6H_4 \cdot NCl \cdot COC_2H_5$, red prisms, m. p. 57° .
Benzoyl-p-aminoazobenzene,—



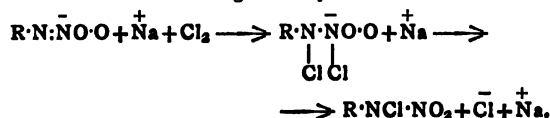
reddish-orange plates, m. p. 211° . *p-Benzoylchloroaminoazobenzene*, $C_6H_5 \cdot N:N \cdot C_6H_4 \cdot NCl \cdot COC_6H_5$, red-brown, glistening, flattened prisms, m. p. 144° .

124. "The Action of Chlorine and Bromine on Nitroaminobenzenes. Part I. *s*-Trisubstituted Chloronitroaminobenzenes." By K. J. P. ORTON.

In the course of an investigation of the conditions under which the nitroamines derived from *s*-trisubstituted anilines (*Proc. Soc. Chem.*, xviii., 111) are transformed into substances containing the nitro-group in the benzene nucleus, the action of chlorine and bromine on these substances was studied.

When chlorine is passed into an aqueous solution of the sodium salt of a nitroamine, or when dilute aqueous bleaching-powder is slowly added to a solution of the nitroamine in acetic acid, the chloronitroamine is formed; thus 1-nitroamino-*s*-trichlorobenzene yields 1-chloronitroamino-*s*-trichlorobenzene, $C_6H_2Cl_3 \cdot NCl \cdot NO_2$.

As the sodium (and other) salts of the nitroamines may probably be represented by the formula $R \cdot N: \bar{N}O \cdot Na$, and, if so, would be the salts of a substituted "imino-nitronic acid" (compare Bamberger, *Ber.*, 1902, xxxv., 54), the author suggested that the reaction of chlorine with the sodium salt might be represented thus:—



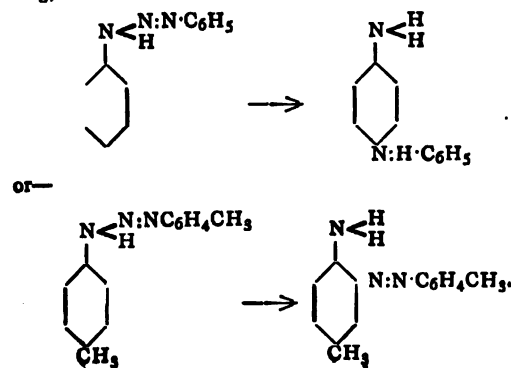
All these chloronitroamines are very unstable, and are re-converted by all reagents into the nitroamines. The following compounds, all of which crystallise in lustrous, colourless prisms, have been prepared:—

1-chloronitroamino-*s*-trichlorobenzene, m. p. $53-54^\circ$; 1-chloronitroamino-*s*-tribromobenzene, m. p. 61° ; 1-chloronitroamino-4-chloro-2:6-dibromobenzene, m. p. 56° ; 1-chloronitroamino-2:3:4:6-tetrabromobenzene, m. p. $61-62^\circ$; 2-chloronitroamino-3:5-dibromotoluene, m. p. 60° ; 4-chloronitroamino-3:5-dibromotoluene, m. p. $50-51^\circ$.

By the action of bromine on aqueous solutions of the sodium salts of the nitroamines, the bromonitroamino-derivatives are formed, but have not been obtained free from the nitroamine.

125. "The Transformation of Diazoamido- into Aminoazo-compounds and of Hydrazobenzene into Benzidine." By F. D. CHATTAWAY.

The transformation of diazoamido- into the isomeric aminoazo-derivatives is similar in nature to the other well-known transformations in which atoms or groups of atoms pass from the nitrogen of an amino-group, attached to a benzene nucleus into the para- or ortho-position in the ring, thus:—



As is usual in these intramolecular changes, the para-position is taken up by preference, the ortho-position to a less extent, or exclusively when the para-position is already occupied.

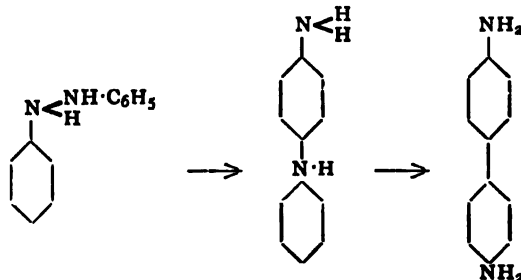
This analogy is somewhat obscured by the ordinary method of representing the transformations,—



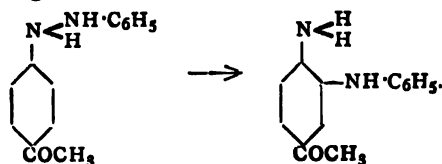
where the NH group appears to leave the chain and pass into the ring.

The formation of the various azo-derivatives which are obtained by heating diazoamido-compounds with aromatic amines and their hydrochlorides, is thus rendered intelligible without assuming the formation of intermediate nitrogen halogen compounds (Goldschmidt and Bardach, *Ber.*, 1898, xxv., 1376). It is only necessary to assume the existence of a mobile hydrogen atom and double linkage in the diazoamido-compounds, and to admit that the diazo-group can undergo transference from the nitrogen atom of one amine to that of another, present in large excess, exactly as a chlorine or bromine atom does. Much that is known of similar isomeric changes points to the conclusion that transference from nitrogen to the ring can only take place when the nitrogen is exerting its higher valency. This may explain the advantage, in these transformations, of the hydrochloride of the base.

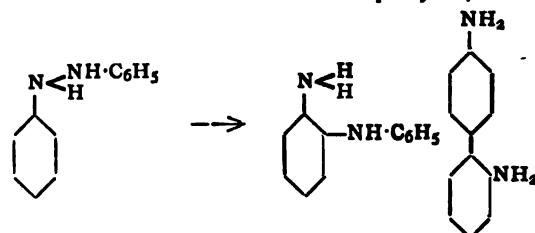
The isomeric change of hydrazobenzene into benzidine is also brought into line with other analogous transformations if similarly formulated:—



It is seen to consist of two stages, of which the first only can take place if the para-position is already occupied, the semidine and other similar transformations resulting:—



As is well known, the related ortho-change also takes to some extent with the formation of diphenylene,—



In all probability, the ortho-change also takes place to a relatively very small extent in the second stage with the production of diorthoaminodiphenyl, though the latter is produced in such small quantity that it has hitherto escaped detection.

126. "Tribromophenolbromide." By E. W. Lewis.

A re-examination of this substance has shown that the melting-point usually attributed to it (118°) is much too low, and that it may be raised to $148-149^{\circ}$ by re-crystallising the compound from ethyl acetate; moreover, whilst, as usually stated, the substance of low melting-point gives off bromine on heating at about 125° , the purified material begins to lose bromine before melting at about 145° .

The crystals of tribromophenolbromide belong to the orthorhombic system, the axial ratios being $a:b:c = 0.7945:1:1.5921$.

NOTICES OF BOOKS.

The New Volumes of the Encyclopædia Britannica. The Third of the New Volumes, being Vol. XXVII. of the Complete Work. London: *The Times*. Edinburgh: Adam and Charles Black. 1902. Pp. 744.

THE Third Volume of this work comprises those subjects which come between CHI (Chicago) and ELD (Elduayen, José de).

There are not many articles in this volume relating to chemical or scientific subjects. The "Condensation of Gases," by Prof. J. D. van der Waals, is treated mathematically and is of some interest. This article is followed by one on the "Conduction of Heat," by Prof. H. L. Callendar; it is divided into nineteen sections, such as—1. Mechanism of Conduction; 2. Law of Conduction; 3. Experimental Methods, such as the Plate method, the Tube method, Forbes's Bar method, the Guard-ring method, &c.; 13. The Periodic Flow of Heat; 15. Annual Variation, &c.

Mr James Douglas contributes an article on "Copper," dealing with its occurrence, metallurgy, and applications. "Diffraction Gratings," by H. A. Rowland, and the "Diffusion of Gases," by G. H. Bryan, are two short articles that will repay reading, while the one on "The Dynamo," by C. C. Hawkins, is of considerable importance and covers no less than twenty pages. "Earthquakes," by J. Milne, will interest many readers, as the subject is one that is attracting a good deal of attention at the present time.

The First Products of the Decomposition of Albumen in the presence of Alkalis.—O. Maas.—The author has endeavoured to find out whether, in the action of alkalis on albumen, one or several albumoses are produced, as in pepsic digestion, or in hydrolysis by weak acids. The solutions used contained 1.60 grms. to 2.25 grms. of sulphur per thousand parts, and were maintained at 15° , 40° , and 90° from one to forty-eight hours. The attack is very rapid, and when the liquid is neutralised it is observed that a mixture of albuminate (acid-albumen) and a new albumose, the *alkali-albumose*, is precipitated. The filtrate contains small quantities of albumose, but no peptone. Ovalbumen and serum-albumen behave in the same manner. The remainder of the paper deals with the description of the new albumose. It is obtained by heating 40 grms. of dry ovalbumen with 1 litre of normal soda for three or four hours on a boiling water-bath. The filtrate is treated with acetic acid so long as a precipitate is produced, and the precipitated mass gives up the new albumose to boiling alcohol. The remainder is precipitated by the addition of water, or, better still, acetone. This body is insoluble in cold water, slightly soluble in boiling water, and fairly soluble in 50 or 60 per cent alcohol. It is no more soluble in saline solutions than in water, but dilute alkalis dissolve it easily. One volume of a saturated solution of sulphate of ammonium precipitates it completely. It gives the Millon, the biuret, the Molisch (α -naphthol), and the Adamkiewicz reactions distinctly, and it blackens an alkaline solution of oxide of lead. It contains:—C, 53.57; H, 7.19; N, 13.62; and S, 2.13 per cent.—*Zeit. Phys. Chem.*, vol. xxx., p. 61—74.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxiv., No. 26, June 30, 1902.

New Researches on Liquid Silicon Hydride, Si_2H_6 .—H. Moissan and S. Smiles.—In a previous paper the authors published an account of the preparation and some of the properties of liquid silicon hydride, Si_2H_6 . They have continued this research, and give further proof of the formula Si_2H_6 . This silicide corresponds to ethane; it is spontaneously inflammable in presence of air, and possesses very energetic reducing properties. It decomposes carbon tetrachloride and sulphur hexafluoride with violence.

Certain Properties of Amorphous Silicon.—H. Moissan and S. Smiles.—When the vapour of liquid hydrogen silicide is decomposed by a series of induction sparks, hydrogen and silicon are obtained. This silicon is in a finely-divided state, and slowly reduces a neutral solution of potassium permanganate at ordinary temperatures. This reduction becomes more rapid at 100° . Various other reductions are effected by silicon prepared in this manner.

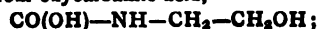
Double Nitrites of Iridium.—E. Leidié.—The author repeats Gibbs and Lang's experiments on iridium nitrites and prepares—(1) The double nitrite of iridium and potassium, $Ir_2K_6(NO_2)_{12}$; (2) the double nitrite of iridium and sodium, $Ir_2Na_6(NO_2)_{12} \cdot 2H_2O$; (3) The double nitrite of iridium and ammonium, $Ir_2(NH_4)_6(NO_2)_{12}$. By means of the chlorides of iridium and nitrites of barium, mercury, and silver, he was not able to reproduce exactly the compounds described by Lang or Gibbs.

Constitution of the Aloines. Comparison with that of the Glucosides.—E. Léger.—In a preceding paper the author showed that barbaloin and isobarbaloin, when acted upon by Na_2O_2 , give the same product of oxidation, methylisoxychryssaine, and that the chlorated derivatives of these same aloines give a single tetrachlorated methylisoxychryssaine. This shows that the two isomeric aloines contain a common radicle. Their properties are very similar; both yield chrysamic acid when treated with HNO_3 . When heated in a dry tube, it gives off vapours which redden aniline acetate paper. A result of the author's researches on this subject show that the aloines belong to a new class of bodies—glucosides which are not decomposed by dilute acids.

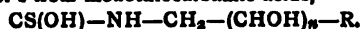
Action of Carbon Disulphide on Polyvalent Amino-alcohols.—L. Maquenne and E. Roux.—Carbon disulphide attacks the polyoxyamines when heated, giving cyclic compounds having a single atom of sulphur; these apparently belong to the family of the oxazolines. These results can be predicted from those obtained by Gabriel with bromated amines and those which were quite recently obtained by Franchimont and Lublin by condensing ethanolamine with methyl chloroformate, and so obtaining the

body $NH \begin{array}{c} \diagup CH_2-CH_2 \\ \diagdown CO-O \end{array}$. Gabriel's compounds result

from the anhydriisation of dithiocarbamic acids, $CS(SH)-NH-CH_2-CHOH-R$; that of Franchimont is derived from oxycarbamic acid,—



the author's from monothiocarbamic acids,—



They consequently form a new series intermediate between the two preceding, and comparable with the substance Hofmann obtained by combining the senevols with the alcohols.

Mechanism of the Synthesis of Leucine.—A. Vila and E. Vallée.—The authors describe a preparation of ammonium-valeral producing a crystalline hydrate. Under the given conditions, the action of hydrocyanic acid on this body yields a single compound, unknown up to the present time, but produced in this reaction in considerable quantity. The hydrolysis of this derivative by dilute sulphuric acid furnishes directly leucine, capable of being sublimed, and characterised by its insoluble copper salt.

MISCELLANEOUS.

Tetrabromide of Platinum.—A. Miolatti and I. Bellucci.—Platinic bromide, $PtBr_4$, is slightly soluble in water, and gives a light reddish-brown solution; the examination of this latter from the point of view of acidimetry, electrical conductivity, and the action of the salts of the heavy metals, leads to the opinion that $PtBr_4$ is the anhydride of a bibasic tetrabromoplatinic acid, $PtBr_4(OH)_2$. The following salts are of a deep brown colour:—Salts of silver, $PtBr_4(OH)_2Ag_2$; of thallium, $PtBr_4(OH)_2Tl_2$; of mercury, $PtBr_4(OH)_2Hg$; and of lead (basic), $PtBr_4(OH)_2Pb.Pb(OH)_2$.—*Zeit. Anorg. Ch.*, vol. xxvi., p. 222.

Action of certain Nitrated Substances on the Coagulation of Albumen.—K. Spiro.—The albuminous solution used was beaten-up white of egg, treated with acid phosphate of potassium until the reaction was acid to litmus; it was then filtered and diluted with varying amounts of water. Mauthner has already shown that choline prevents the coagulation of ovalbumen. Piperidine exercises the same action. Bases, such as pyridine and aniline, which are precipitants of albumen, are able, nevertheless, to stop a portion of the albumen from coagulation. Orthotoluidine (but not the para derivative) and xylydine act in the same manner. Nitrated substances of a non-basic character, such as urethane, various amides (formamide, urea), and the senevols, have the same action. The explanation of this action must be sought for in the basic character of these substances, a character strongly marked in some of them, but less apparent in others. But Ostwald has remarked that we can attribute to urea in solution a formula such as $CO.NH_2.OH.NH_2$, and, on the other hand, the author shows that albumen, held in solution by concentrated solutions of urea, takes all the characteristics of an alkali-albumen, and that in the coagulation of ovalbumen the urea acts decidedly as an antagonist to acetic acid.—*Zeit. Phys. Ch.*, vol. xxx., p. 182—199.

Pentachloroplatinic Acid.—A. Miolatti and I. Bellucci.—The authors have examined the bodies which result from the substitution of 1, 2, 3, &c., hydroxyl for an equal number of atoms of chlorine in chloroplatinic acid (hexachloroplatinic), $PtCl_6H_2$, and are at present at work on the first term of the series, pentachloroplatinic acid, $PtCl_5(OH)H_2$, which is no other than the product $PtCl_4.HCl.2H_2O$, prepared by M. Pigeon (*Ann. Chim. Phys.*, Series 7, vol. ii., p. 433), by heating chloroplatinic acid to 100° for two or three days in an exhausted tube containing a few fragments of potash. On repeating this experiment on a larger scale in an exhausted oven with a larger surface, and in which the potash could be renewed during the operation, they easily obtained M. Pigeon's product in the form of a reddish-brown mass, fusible on the water-bath, extremely deliquescent, and giving yellowish-brown slightly cloudy solutions with a distinctly acid reaction. These solutions are not precipitated in the cold by ammonia, which is distinctive of $PtCl_6H_2$; with KCl or NH_4Cl we get the ordinary chloroplatinates. Acidimetry and the study of the electric conductivity show that we have to deal with a bibasic acid of which several salts have been prepared. Salt of Barium, $PtCl_5(OH)Ba_2.4H_2O$.—The acid is exactly neutralised by

baryta in concentrated solution; it is then evaporated in the desiccator, and gives long orange-coloured prisms. Salt of Silver, $PtCl_5(OH)Ag_2$.—Prepared from nitrate of silver in the cold, an abundant yellowish precipitate is formed, not decomposable by boiling water; this distinguishes it from the ordinary chloroplatinate, $PtCl_6Ag_2$, and shows that it is nearer the tetrachloroplatinate, $PtCl_4(OH)_2Ag_2$. Salt of Thallium, $PtCl_5(OH)Tl_2$.—This is a pale rose-coloured precipitate. Basic Salt of Lead, $PtCl_5(OH)Pb.Pb(OH)_2$.—Yellowish precipitate.—*Zeit. Anorg. Ch.*, vol. xxvi., p. 209.

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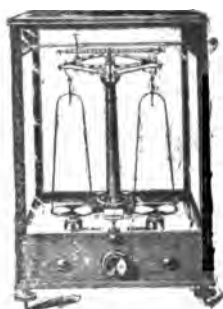
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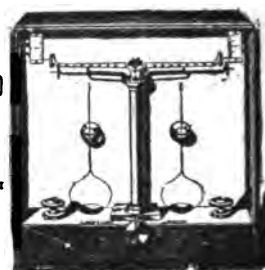
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THE CHEMICAL NEWS.

VOL. LXXXVI., No. 2227.

A NOTE ON THE RE-CRYSTALLISATION OF PLATINUM.*

By WALTER ROSENHAIN, B.A (Cantab.), B.C.E. (Melbourne).

IN a recent paper ("On the Crystalline Structure of Metals," second paper, *Phil. Trans.*, A, 1900, vol. cxcv., pp. 279-301) Professor Ewing and the present author have described phenomena of re-crystallisation in a number of metals, such as lead, tin, zinc, and cadmium, at temperatures well below the melting-points of those metals. I have recently observed phenomena which appear to me to be of a very similar nature in the case of platinum.

blowpipe flame. A closer examination revealed a multitude of geometrical pits (*Aetzfiguren*) clearly demonstrating that the appearance is due to genuine metallic crystals which have been *etched* on the surface by some chemical agent. Two other experimental facts confirm this idea. The first is that if a piece of platinum showing the "changed" surface be exposed to the action of aqua regia, the appearance is intensified and brightened. If, as has been suggested, the appearance were due to a superficial layer of a platinum carbon compound from which the carbon had been driven off, leaving mere pseudomorphs of platinum, the etching action of the aqua regia would have dimmed the appearance instead of brightening it. Another objection to the carbonisation theory is to be found in the fact that I have produced the effect on a *new* platinum crucible by prolonged heating in an oxygen injector furnace, where an oxidising atmosphere was being maintained.

The "changed" platinum is, moreover, very weak and brittle when hot, and on one occasion a crucible was torn whilst still red hot, but after being removed from the flame. The fracture was as crystalline as that of



FIG. 1.—SURFACE OF A PLATINUM CRUCIBLE AFTER PROLONGED HEATING.
(Oblique Light. Magnification, 30 diameters).

It is a well-known fact that a prolonged exposure to a high temperature renders platinum brittle, and that the surface of such platinum, when it has been exposed to flame, shows markings "resembling the appearance of galvanised iron" ("The Action of the Acetylene Flame on Platinum," J. J. Redwood, *Journ. Soc. Chem. Industry*, vol. xviii., p. 1107; also *Zeit. für Analyt. Chem.*, 1901, Heft 6, p. 411). This phenomenon has generally been ascribed to the action of carbon, and by one author specifically to the action of acetylene (*Ibid.*). Having studied the phenomena closely with the aid of the microscope, I do not find this view entirely confirmed.

In the first place, on examining the surface of the "changed" platinum with the microscope, it is seen to show a pattern which is characteristic of the *etched* surface of a crystalline metal. The micrographic appearance under oblique light is shown, magnified 30 diameters, in the photograph (Fig. 1). This was taken from the surface of a platinum crucible which has been in continuous use, and is frequently exposed for hours together to an ordinary

"brittle" zinc, and the lines of fracture ran across the crystals revealed on the changed surface in such a way as to show that these crystals seen on the surface are not a thin superficial layer, but genuinely represent the entire structure of the metal.

I am therefore led to the conclusion that the action is simply one of re-crystallisation. The metal in the state in which it reaches us in foil or crucibles, &c., is in a condition of severe strain, having been bent, drawn, rolled, &c., either in the cold or at temperatures far below its "annealing" temperature. This is supported by the fact that the platinum in its "unchanged" state shows a very minute structure characteristic of severely strained metals. The natural effect of exposure to a high temperature of metal in such a condition is to allow it to re-crystallise, and this I conceive to be what occurs in the case of platinum. The brittleness of the "annealed" metal is not at all surprising, as the same phenomenon occurs with zinc and cadmium (see paper cited above). In the case of platinum "annealed" in a gas flame there is, however, a further action; simple annealing or re-crystallisation, although it will completely alter the interior structure of

* A Paper read before the Royal Society, May 15, 1902.

a piece of metal, will not of itself alter the appearance of the surface even in microscopic detail. To develop a surface pattern corresponding to the changed internal structure the surface must be etched after the re-crystallisation has taken place. The etching action is in this case undoubtedly due to the gases of the flame, and the temporary formation of a carbide may play a part in this process.

SOME REMARKS ON A. BACH'S PAPER,
"THE MECHANISM OF THE ACTION OF
PEROXIDE OF HYDROGEN ON
PERMANGANIC ACID,"

IN SO FAR AS IT INVOLVES THE QUESTION
OF THE VALENCY OF HYDROGEN.

By GEOFFREY MARTIN, B.Sc.(Lond.).

WITH regard to the main part of the paper (*Moniteur Scientifique*, January, 1902, Series 4, vol. xvi.) I have nothing to say. It involves a point that must be settled by experiment.

But I take exception to his remark:—"To admit, as is done by Traube, that peroxide of hydrogen contains easily oxidisable hydrogen united directly with active oxygen seems contrary to reason."

For my part I fail entirely to see that such an assumption is contrary to reason. It is true that it involves the supposition that a hydrogen atom can combine with more than one atom at the same time—in other words, can act as a polyvalent element. But in support of this view there is a great body of experimental facts slowly accumulating.

A brief review of this evidence will perhaps be useful, as outside a special circle the majority of chemists still seem to regard hydrogen as the stock instance of a monovalent element.

When a hydrogen atom enters into combination with an atom more negative than itself, such as O or Cl, it almost invariably acts as a monad.

At the time the valence theory was broached these were practically the only class of hydrogen compounds known, and it was for this reason that this element was labelled "monad."

But an entirely new class of compounds have within recent years come to light. They are produced by the union of hydrogen with elements more positive than itself; and in these compounds it no longer acts as a monad, but appears often to be playing the part of a polyvalent element.

The following are a few of these compounds:—

Na_2H .—Soft wax-like substance, formed at 300° and dissociated at 421° . (Troost and Hautefeuille, *Comptes Rendus*, lxxviii., 807).

K_2H .—White saline crystalline compound. (Raoult, *Comptes Rendus*, lxi., 826).

(In these two compounds the minimum value we can ascribe to H is 2).

$\text{FeH}_2(?)$.—Black powder, like finely divided iron; evolves H on heating and on treatment with H_2O . Formula conjectural, as it has not yet been analysed. (Ramsay, "System. Chem.," 1891, p. 575).

Pd_2H .—According to Ramsay (*loc. cit.*), "The alloy approximates in composition to that expressed by the formula Pd_2H , but it appears to absorb hydrogen in excess, which is not chemically combined, but in a state of mixture." Now palladium only forms two classes of compounds—the palladous, in which it is divalent, and the palladic, in which it is tetravalent. Therefore the lowest valency H can have here is 4 and the highest 8.

NbH .—Black powder. (Krüss, *Ber.*, xx., 169). Since niobium acts invariably as a quinquevalent element, the value H can have here is 5.

Vd_3H_2 .—Vanadium can act as a monad, dyad, triad, or pentad element. If it acts as a monad in the above compound the valence of H would not be a whole number. If it acted as a dyad, the value of H comes out as 3.

CuH .—Black powder, decomposes between 55° and 60° . (Wurtz, *Annales*, [3], xi., 251). If Cu be here in a cupric state, H is divalent; if in a cuprous state, H is monovalent.

Traces are met with of a great many others, but in the majority of cases they are very imperfectly known.

It would appear that in them hydrogen can act respectively as a monad, dyad, triad, tetrad, and pentad element.

The fact that these compounds are unstable and rare has nothing whatever to do with the validity of the conclusions to be drawn from the behaviour of hydrogen in them.

Under other conditions, a rare and unstable compound may become common and stable, and a stable common compound rare and unstable. For instance, were there little oxygen on the earth, H_2O would be a very rare compound; and did we live at a temperature of 4000°C ., it would, in addition, appear to us as an extremely unstable body, easily decomposable into H and O gases. In fact, under such conditions, it would rank in our estimation as do some of the hydrides at ordinary temperatures. But its scientific significance would be quite as great under these as under normal conditions.

Similarly, did we live in an atmosphere of hydrogen—such as exists in some stars—and under a great pressure and temperature, the hydrides would probably be very prominent and abundant bodies.

Their existence, therefore, cannot be ignored; and the rôle of hydrogen in them demands an explanation with as great a force as if they were the most prominent section of the hydrogen compounds.

In this connection it is interesting to note that definite halogen compounds of potassium, rubidium, and cesium are known in which the metal plays a monad, triad, and pentad rôle.

Thus, of the type RX_3 , we know—

KI_3 ;
 RbBr_3 , RbI_3 , RbClBr_2 , RbCl_2Br , RbBr_2I , &c.;
 CsBr_3 , CsI_3 , CsClBr_2 , CsCl_2Br , CsClBrI , &c.;

and of the type RX_5 , we have—

KICl_4 ;
 RbCl_4I ;
 CsBr_5 , CsI_5 , CsCl_4I .

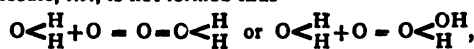
Towards oxygen and sulphur they also appear to be able to act as divalent and tetravalent elements.

It will not be, therefore, very surprising if hydrogen, which belongs to the odd series of Group I., is also found to be capable of acting as a polyvalent element in certain rare cases.

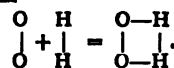
If we assume that in hydrogen peroxide it is acting with a valency of 2, and that this compound has the constitution

$\text{O}-\text{H}$
 $\text{O}-\text{H}$, we get a perfectly clear interpretation of the fact,

so ably demonstrated by Traube, that hydrogen peroxide is not an oxidised water molecule, but a reduced oxygen molecule, i.e., is not formed thus—



but is formed thus—



It may be noted that it is almost certain, both from its physical and chemical behaviour, that this compound does not contain hydroxyl groups.

We have pointed out that when electro-positive hydrogen enters into combination with an electro-negative element, it usually acts as a monad; but when it combines with an element of positive polarity, it appears to behave as a polyvalent element,

This appears to be quite a general phenomena. For instance, when the negative halogens combine with positive radicals, they invariably act sharply as monads. But when they enter into combination with radicals of like sign, they no longer act as monads, but exhibit a polyvalent nature, e.g., in ICl_3 , $\text{I} \begin{matrix} \text{C}_6\text{H}_5 \\ \text{C}_6\text{H}_5, \text{ \&c.} \\ \text{OH} \end{matrix}$.

The metals, too, when they unite with each other, give rise to phenomena of an essentially similar nature. They appear to act towards each other with a quite different valence to that with which they usually act towards negative radicals. For instance, there are known to exist definite compounds of the formulæ AuCd , PtAl_3 , Au_4Pb , Au_3Pb_2 , SnSb , and many others much more complex (see Nevill, "Report on the Nature of the Compounds contained in Alloys," British Association, 1900).

So that if it is ultimately found that electro-positive H enters into union with an electro-positive element, it will at least have a precedent in the case of actually known elements.

A. Bach's assertion, therefore, that "to admit that . . . hydrogen peroxide contains easily oxidisable hydrogen united directly with active oxygen seems contrary to reason," is by no means self-evident; but, on the other hand, it appears that the facts speak strongly for the view that in certain cases hydrogen is capable of combining with more than one atom at the same time.

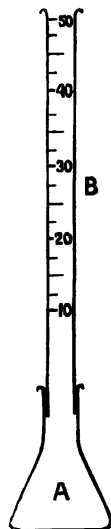
University of Berlin, May 26, 1902.

APPARATUS FOR DETERMINING MINERAL OIL IN A MIXTURE OF MINERAL AND VEGETABLE OILS.

By R. F. YOUNG and B. F. BAKER.

We beg to submit the accompanying apparatus for rapidly and accurately determining the per cent of mineral oil in a mixture of mineral and vegetable oils.

Into the flask A is ground an accurately graduated glass tube, holding about 60 c.c. of liquid and graduated to one-



tenth c.c. To use the apparatus 50 c.c. of the oil under examination is measured into A, excess of alcoholic potash added, and the oil saponified by heating on the water-bath in the usual way with a reflux condenser or simply a long glass tube, which may also be ground to fit

A. When saponification is complete, the flask is fitted with B, and water added till the unsaponified oil rises into the graduated tube. The volume of oil is then read off. Then $\frac{\text{Volume of oil in tube}}{50} \times 100 = \text{per cent of mineral oil in sample.}$

The Laboratory,
Wouldham Cement Company (1900),
West Thurrock, Essex.

NOTES ON COCOA ESSENCES.

By E. G. CLAYTON, F.I.C.

THERE are very few published determinations of the cocoa-starch or of the caffeine, as well as theobromine, present in "cocoa essences" (cocoas deprived of a part of the fat, and otherwise treated). The following analyses, which in most cases include these details, may therefore be useful for reference:—

Numbers 4 and 5 contained minute quantities of foreign starches, so that the starch in the former was nearly—but not quite—all cocoa-starch. Some of the caffeine also in No. 4, and the maltose, were extraneous. Considerable variations are observable in the proportions of cellulose and soluble alkali in these preparations of cocoa. Bell's process was used for the determinations of theobromine and caffeine; and starch was determined, after removal of the fat, alkaloids, and carbohydrates soluble in cold water, by conversion into dextrose and titration with Fehling's solution (10 dextrose = 9 starch). Protein nitrogen was Kjeldahlised after extraction of the alkaloids.

	Sample.				
	1.	2.	3.	4.	5.
Water	4.62	3.22	3.59	5.33	4.28
Fat	33.11	32.69	30.50	22.30	27.46
Theobromine	1.82	0.93	0.88	0.83	2.69
Caffeine	0.08	0.02	0.42	0.66	0.16
Starch	5.63	6.56	—	5.06	—
Proteids	15.56	14.31	14.19	19.50	12.12
Maltose	—	—	—	1.64	—
Cellulose	5.65	9.29	6.97	5.59	4.21
Cocoa-red, dextrin, tannin, &c.	27.82	25.15	37.35	32.47	42.77
Ash	5.71	7.83	6.10	6.62	6.31
	100.00	100.00	100.00	100.00	100.00
Soluble ash	3.20	7.44	5.67	4.67	3.13
Insoluble ash	2.51	0.62	0.43	1.81	3.18
Alkalinity (as K_2O) of soluble ash	1.16	2.94	2.21	1.48	0.86
Total phosphoric acid (P_2O_5)	1.91	1.72	1.93	1.92	1.29
Soluble phosphoric acid	—	—	—	—	1.15
Insoluble phosphoric acid	—	—	—	—	0.14
Silica	—	0.36	—	0.69	—
Iron oxide	—	trace	—	0.32	—
Proteid nitrogen	2.49	2.29	2.27	3.12	1.94
Cold water extract (total)	17.90	17.20	14.56	18.25	—
Cold water extract (organic)	15.10	12.30	10.48	13.29	—
Alkalinity of cold water extract	2.76	3.39	2.72	2.75	—
Ash of cold water ex- tract	2.80	4.90	4.08	4.96	—

Chemical Laboratory,
32, Holborn Viaduct, London, E.C.,
July 15, 1902.

ON RADIO-ACTIVE BISMUTH (POLONIUM).
(PRELIMINARY COMMUNICATION).

By W. MARCKWALD.

IN the year 1898 P. and S. Curie were induced, by Becquerel's beautiful observations on the peculiar radiation of uranium minerals and preparations, to submit the pitchblendes to a searching chemical examination. Thus they found, in extremely small quantity in these minerals, two exceedingly interesting constituents, which were radio-active in a far higher degree than the uranium compounds.

The substance which was first discovered gave all the chemical reactions of bismuth, and differed from it only in radio-activity, which was about a hundred times that of pitchblende. By partial sublimation of the sulphide, partial precipitation of the nitrate by means of water, &c., the metal, in the form of the compounds in question, could be decomposed into inactive bismuth derivatives on the one hand, and, on the other, into about four times as much of compounds of radio-active bismuth. Although the discoverers had no doubt that the final product consisted essentially of bismuth only, they did not succeed in increasing the proportion of the active constituent. They suggested Polonium as the name of the unknown element which was supposed to be the cause of the activity.

The discovery of radium soon followed that of radio-active bismuth. The authors were more fortunate in their attempts to separate radium from barium. For, by crystallisation of the chloride, they succeeded in freeing the radium almost entirely from the barium, though they could not obtain it perfectly pure. I have recently described how, by a specially adapted method of crystallisation, the separation of the barium from the chloride containing radium can be brought to any degree of perfection.

The further examination of these radio-active substances, to which were soon added others—actinium, europium, radio-active lead,—brought to light a striking difference between the activity of radium and that of polonium, namely, while the activity of freshly separated radium preparations at first increased strongly and finally became constant, F. Giesel observed that his radio-active bismuth, for the most part, lost its activity after some weeks, and Curie's preparation exhibited the same phenomenon, though more slowly. This led Giesel to the supposition that, "in polonium, we have before us probably nothing more than bismuth made active by induction." For it has been proved repeatedly that radio-active substances can transfer their activity to other substances, especially if the latter are precipitated from a solution containing active substances.

The discoverers of polonium lately seem to have adopted Giesel's opinion. At any rate, an article by P. and S. Curie which has recently appeared contains in a footnote the sentence:—"Polonium is a species of active bismuth; there is as yet no proof that it contains a new element."

Observations which I have made during the examination of radio-active bismuth have already elucidated in some respects the nature of this substance. In order to secure to myself the undisturbed continuation of these experiments, the preliminary results are here shortly described.

In the autumn of last year some kilograms of a by-product obtained from pitchblende were sent to me for examination by the firm of Dr. Richard Sthamer, of Hamburg. According to information supplied to me, the material formed the residue which remained after treatment of pitchblende with concentrated sulphuric acid and washing with water. The aim of the research was first to determine whether radio-active substances could be obtained from the material, and finally to work out a method which could be used technically for the separation of these substances.

The material was comparatively rich in radio-active bismuth. By already known methods, about 1 per cent by weight of bismuth oxychloride could be obtained from the raw material. The product was strongly radio-active. No decrease in the activity has been observed after the lapse of several months.

Many attempts were made to separate a radio-active constituent from this substance; some were unsuccessful, and some gave unsatisfactory results, which still remain to be further investigated. Finally, the problem was successfully solved by a very simple method.

Starting from the assumption that, in radio-active bismuth, besides ordinary bismuth, a second radio-active metal is present, which very closely resembles the first in its chemical reactions, the behaviour of the salts on electrolysis was examined. If the two metals exhibited any difference of potential whatever—and this was to be expected from the great analogy in chemical behaviour—this must become apparent on electrolysis, so that either the active or inactive constituent would increase in the metal which first separated out. An experiment was performed under quite arbitrarily chosen conditions, and the result showed that the metal which separated out first exhibited a much greater activity than the final product. This result led to an attempt to directly replace in the hydrochloric acid solution of the chloride the ions of the radio-active metal by bismuth ions, by immersing a polished stick of bismuth in the solution.

As was to be expected, the metal became coated immediately with a fine black deposit, which increased visibly in a few hours. When taken out of the solution and washed with hydrochloric acid and alcohol, the stick had a surprisingly strong effect on the electroscope. At a distance of a decimetre the leaves of the charged electroscope collapsed in a moment, and even a gutta-percha rod which had been well rubbed with fur was at once discharged on the approach of the bismuth stick.

The fact that all the radio-active metal is deposited on the bismuth stick in the course of a few days is of the greatest importance. When 8 grms. of bismuth oxychloride, in a hydrochloric acid solution, were allowed to stand for three days with a small plate of bismuth, the salt in the solution was practically inactive. To confirm this a new stick of bismuth was left in this solution for twenty-four hours; it remained perfectly clean, and was only very slightly radio-active.

The deposit could be easily and almost entirely scraped off the bismuth plate; it weighed about 5 m. grms. Hence radio-active bismuth contains probably at the most one-tenth per cent of the radio-active metal. Therefore a ton of pitchblende would contain roughly not more than 1 gm.

The substance is not the pure metal, but contains some chlorine. On heating, a small portion volatilises, probably in the form of chloride. The residue fuses to a white shining metallic bead, which is exceedingly brittle. It is easily soluble in nitric acid. As far as the solution has yet been examined for bismuth, it gives the characteristic reactions of that metal. Nevertheless, the metal cannot be identical with bismuth, for the separation of a metal of its own accord from a solution would violate the law of conservation of energy, except in cases of the formation of concentration currents, which is impossible in this instance.

The rays emitted from the metal, and equally strongly from the salts as well, are characteristically different from those of radium, for they seem unable to penetrate any intervening substance. If a stick of bismuth, prepared according to the above method and having a strong effect on the electroscope, be merely lightly wrapped in filter-paper, it loses all its effect.

This feeble power of penetration of the rays is also the cause of the fact that no appreciably stronger effects are obtained by the use of thicker deposits than those of about a ten-thousandth of a gm. The firm of Dr. Richard Sthamer intends to bring into the market such little sticks for use in physical researches and demonstrations.

My next task will be to obtain enough of the new metal to carry out an atomic weight determination. The above-named firm, which has most kindly done much to facilitate my researches, has, by following my instructions, obtained in their factory bismuth from pitchblende in large quantity, and also radium; and I owe them my hearty thanks for sending me almost 1 kilo. of bismuth oxychloride. I hope to be able to attain the desired end with this quantity.—*Berichte*, 1902, xxxv., 365.

EXPERIMENTAL RESEARCHES ON THE CONSTITUTION OF CRYSTALS.*

By A. E. TUTTON, B.Sc., F.R.S., F.C.S.

(Concluded from p. 43).

THERE will now be introduced to you another means which we possess of determining the position and shape of the ellipsoid. In an ellipsoid having three unequal rectangular axes, it will be evident that if we consider the elliptical section containing the maximum and minimum axes, there must be a point somewhere on each elliptical quadrant where the radius vector will be equal to the intermediate axis. Such are the four points C on the figure projected on the screen. The two sections of the ellipsoid which contain these points and the intermediate axis will consequently be circles, and rays of light which pass through the crystal at right angles to these sections will be able to vibrate with equal velocity in all directions perpendicular to the line of propagation. Consequently in these two directions, which are termed optic axes, the crystal will exhibit no double refraction. When the Y velocity approaches nearer to the X velocity the inclination of the two circular sections to each other naturally diminishes, and the angle between the optic axes becomes proportionally less, until at length we have equality of X and Y, the ellipsoid becoming a spheroid, with a single optic axis. This is the special case which we observe in tetragonal and hexagonal crystals, such as the well known cases of calcite and quartz.

We will now see some very beautiful phenomena in connection with these optic axes, employing for the purpose a projection polariscope, which is furnished with a special set of lenses to render the light strongly convergent while passing through the crystal. First let us investigate the phenomena exhibited by one of the uniaxial crystals we have just referred to. A section-plate cut perpendicular to the single optic axis is now between the crossed Nicols, and you see the beautiful spectrum rings and the black cross, characteristic of uniaxial crystals, which are produced.

We will next see the effect produced by a section-plate of a biaxial crystal, similar to the salts we are discussing. The plate is cut perpendicular to that axis of the ellipsoid which is the bisectrix of the acute angle between the optic axes. You observe the loci of the two optic axes are marked by hyperbolic brushes, in the present position of the section, and they are surrounded by separate rainbow-coloured rings, which in turn are surrounded by lemniscates, and eventually ellipse-like curves. If we rotate the section 45°, the hyperbolæ join up to form a cross, but the loci of the optic axes remain marked by the rings. These rings are large and brilliant, to render the demonstration clear, and are afforded by a very large section-plate. But for research purposes we require the rings to be very small and the hyperbolæ very narrow, so that the measurement of their position may be very accurate.

We will put in another section-plate, much smaller because of the impossibility of obtaining larger ones, of rubidium magnesium selenate, and you see how small and sharp, although naturally fainter, the rings and hyperbolæ are.

Next let us demonstrate how we measure the angle of separation between the two optic axes. Another section-plate has been arranged on a small goniometer, so that it can be rotated in the plane of the axes. You see we are able to bring first one and then the other optic axis up to the cross wires, which you see also focussed on the screen.

If we note the reading of the goniometer circle while one optic axis is so adjusted, and then rotate until the other is in position and read the circle again, the difference between the two readings will give us the apparent angle between the axes as seen in air.

In order to arrive at the true angle within the crystal, it is necessary to cut another section perpendicular to the bisectrix of the obtuse angle between the axes, and to measure this obtuse angle. As, however, this large angle is usually invisible in air, owing to internal reflection, it is necessary that both angles shall be measured while the sections are immersed in some highly refractive liquid. A simple calculation, involving the two angles measured, enables us then to deduce the true optic axial angle within the crystal.

The result of a large number of such measurements of optic axial angles has been to show that in all cases where the interference figures are normal, the angle of the rubidium salt is intermediate in value between the angles of separation of the optic axes of the potassium and caesium salts of the same triplet.

We will conclude the lecture by demonstrating to you the beautiful optic axial phenomena in the four abnormal cases to which reference was made when discussing the refraction phenomena, in which, for a certain wave-length of light a prism of the crystal only gives one refraction image instead of two.

You see on the screen the type of interference figure which is given by rubidium sulphate. It is characteristic of the few known interesting salts which exhibit the phenomenon of crossed axial plane dispersion.

You see next the interference figure afforded by caesium magnesium selenate. It is characterised by large dispersion, and it will be proved to you in a moment that for blue light this crystal also is apparently uniaxial, and that the figure in white light which you are now contemplating is due to the fact, that for the most luminous colours of the spectrum separation of the optic axes in the horizontal plane occurs.

It would be interesting to analyse this figure by showing you the curves afforded by the crystal in the pure monochromatic light from the spectroscopic illuminator. But although this can be done most brilliantly for one person at a time looking through the so illuminated observing instrument, there is not light enough for projection. But a series of six photographs, for six specific wave-lengths of light, have been taken with the aid of the apparatus, and you now see them on the screen, each with as exact a reproduction of the colour for which it was taken as possible. The first shows the separation for red lithium light, the second the diminished angle for yellow sodium light, the third the still smaller angle for green thallium light, the fourth the further approach towards the centre for greenish-blue hydrogen light, the fifth shows the uniaxial figure in light of wave-length 466 for which crossing occurs, and the sixth shows the separation in the perpendicular plane for violet hydrogen light.

We will also show you another six, to prove to you that not only does change of wave-length in the illuminating light provoke extraordinary changes in the optic axial angle, but that change of temperature is also provocative of remarkable changes of angle. This set represents the phenomena observed at about 80°. The angle for lithium light is now very much smaller than it was at the ordinary temperature; for red hydrogen light it is still smaller, and for sodium light the crossing has now actually arrived, while for thallium green, hydrogen greenish-blue, and the blue light for which crossing occurred at the ordinary temperature, the axes are separated at increasing angles in the perpendicular plane.

* A Lecture delivered at the Royal Institution of Great Britain, Friday, May 2, 1902.

Our last experiment will illustrate the effect of change of temperature in the case of cesium selenate. The behaviour of this salt is so similar to that of the long-known case of gypsum, that, as I can obtain a very much larger crystal of this beautiful mineral, suitable for projection purposes, I shall use it to demonstrate the phenomena. You see at the ordinary temperature nothing whatever beyond the fact that some light gets through to the screen. The optic axes are at present separated in the horizontal plane to such an extent that they are well outside the field. We are now warming the crystal, and you see colour making its appearance at the sides; now the axes themselves, surrounded by their coloured rings, are appearing. They approach the centre, they now coalesce to form the uniaxial cross and spectrum circles. They now separate in the vertical plane, and we remove the source of heat, the axes still continuing to separate vertically until the crystal and the metal frame in which it is being heated have taken up the same temperature. Now the motion stops, and the phenomena repeat themselves in the inverse order, once more coming to a cross and circles, and again separating in the horizontal plane, until finally they disappear on the margin of the field.

These beautiful cases of crossed axial plane dispersion, you will remember, are entirely due to the operation of the rule which we found to govern the progress of the double refraction, in accordance with the progress in the atomic weight of the alkali metal, so that these very exceptional cases are in reality strong proofs of the main generalisation derived from this work.

It has thus been amply demonstrated to you that the chief result to which these researches have led is, that the members of every series of salts known to the chemist, which differ by containing different elements of the same family group, exhibit perfectly regular variations in their exterior morphology and in their interior physical properties; and that these variations follow the order of the differences between the atomic weights of those interchangeable elements. Thus, it has been shown that the crystallographical properties of the elements are in line with all their other properties, chemical and physical, in exhibiting the same progressive character which is so conveniently expressed by their atomic weights.

We do not yet know why a particular series of salts chooses the specific type of symmetry which is common to its members, but we have reasonable ground for hope that further work in the direction indicated, together with a successful development of the interesting mathematical and geometrical researches now being conducted by several fellow-workers, on the possible modes of partitioning space and the types of molecular packing, will eventually lead to a solution of this important question.

During the researches described in this lecture many thousands of crystals have had to be prepared in a state of perfection and purity, many hundreds of section plates and prisms cut and ground, and innumerable measurements with the most refined of instruments carried out. But one forgets the labour in the contemplation of knowledge truly gained, and one remembers only the delights of the way, the glorious phenomena of colour which one has enjoyed, and the exquisite beauty of the crystals themselves.

ON AMMONIO-CALCIC PHOSPHATE.

BY HENRI LASNE.

I FIRST obtained ammonio-calcic phosphate in 1894, and I mentioned it incidentally in a communication made to the Société Chimique in December, 1896, on questions relating to the analysis of phosphates, but I did not lay much stress on the subject.

My attention has been recalled to this matter by a paper by M. Barthe (*Bull. Soc. Chim.*, Series 3, vol. xxiii., p. 422) on the ammonio-earthly phosphates, in which, after

having referred to Kippenberger's statements, and pointed out their errors, he describes a number of negative experiments, and concludes that the only ammonio-magnesian phosphate exists among the alkaline-earthly ones.

I can only confirm the results of these experiments, since we do not easily find the ammonio-calcic phosphate under the conditions arranged by M. Barthe. This body is fairly soluble in, or at least easily decomposed by, water, and the complete insolubility of the tri-calcic phosphate causes the precipitation of the latter, which is obtained by itself as soon as the solution of phosphate of lime is made alkaline by ammonia, no matter what other precautions may be taken.

It is no longer the same, however, if we prevent the precipitation of the tri-calcic phosphate by means of citric acid, and in this case we are able to obtain the ammonio-calcic phosphate.

This is what happens if we treat a solution of phosphate of lime free from magnesia, with citrate of ammonia and ammonia. When the temperature is brought down to about 6° we obtain fairly large and brilliant crystals which consist of ammonio-calcic phosphate. It is in this manner that I obtained this body for the first time, by accident, while trying to determine whether the magnesia could be estimated in a phosphate by direct precipitation.

In the crystals obtained I was able to detect the presence of ammonia, and after calcination I found:—

Lime	0.1882
Phosphoric acid.. .. .	0.2265

which is in theoretical proportion.

Further, in the uncertainty in which I was at this time as to the exact nature of the body obtained, I weighed the lime in the form of carbonate and of sulphate for the purpose of verifying its identity, and I found:—

Carbonic acid	0.1482
Sulphuric acid	0.2660

which represents the correct equivalent of these acids for the above mentioned quantity of lime found.

I have recently repeated these experiments, taking an excess of pure carbonate of lime dissolved in hydrochloric acid and pure phosphate of ammonia. This mixture was treated with a quantity of citrate of ammonia sufficient to keep the phosphate of lime in solution in a strongly ammoniacal solution. The solution was exposed to a temperature of about 4° to 6°, during the night, and hard, transparent crystals were obtained of a size easily discernible by the naked eye.

In this first attempt I did not dare to wash these crystals thoroughly for fear of seeing them disappear, as I was already aware of their easy decomposition, and, for the same reason, I simply pressed them between sheets of blotting-paper after a slight washing with water containing one-third ammonia before proceeding with their analysis.

In a second experiment I slightly increased the quantity of phosphate of ammonia, and made the solution much more ammoniacal in the hope of obtaining an increased return, but under these conditions nothing but triammonic phosphate was separated, almost free from lime. The very slight solubility of the triammonic phosphate in the ammonia concentrated to half the total volume is a danger which must be guarded against.

To obtain larger quantities of the product it is advisable to dissolve 10 grms. of pure carbonate of lime in hydrochloric acid, then add citric acid so that the ammonia will not give any precipitate in the presence of phosphoric acid. About 15 grms. of citric acid are required for each gramme of lime.

To this solution, made slightly alkaline with ammonia, we add every day a little phosphate of ammonia in concentrated solution, and ammonia, and leave the whole each night where the temperature will fall to about 6°.

These progressive additions enable us to obtain a sufficient quantity of the ammonio-calcic phosphate to carry

out a proper examination, by almost completely avoiding the separation of the triammonic phosphate; wash with water containing one-third of ammonia, then drain and leave the crystals in the desiccator for two or three days.

The products obtained in the first experiment and in the last have been analysed. The phosphoric acid was weighed as pyrophosphate of magnesia, after precipitation by ammoniacal chloride of magnesium in the presence of citrate of ammonia and ammonia in excess; the lime was estimated in the form of sulphate separated by alcohol, and the ammonia by distillation. The loss at 100° was also estimated as well as the loss by calcination before the blowpipe. These analyses led to the formula $\text{PO}_4\text{Ca}_2\text{NH}_4\cdot 7\text{H}_2\text{O}$.

The following are the results compared together:—

	Theory.	I.	II.
Phosphoric acid..	25'45	25'50	26'09
Lime	20'07	19'09	19'79
Loss at red-heat ..	54'48	55'24	54'01
	100'00	99'83	99'89
Nitrogen	5'02	6'98	5'30
Loss at 100°	—	48'27	47'96

We thus found an excess of phosphoric acid and nitrogen easily explained by what has been observed with regard to the almost complete insolubility of the triammonic phosphate—an excess which is more noticeable in No. 1, as this sample was only slightly washed and contained ammoniacal water held in the interstices. The variations being much less with No. 2, which was more thoroughly washed and dried, it is safe to admit that if the washing with ammoniacal water and the drying were better done we should get at the composition with greater accuracy.

It is easy, in fact, to calculate that these products contain:—

	I.	II.
Ammonio-calcic phosphate ..	95'02	98'60
Triammonic phosphate ..	2'62	—
Biammonic phosphate ..	—	1'40
Ammoniacal water	2'34	—
	100'00	100'00

During the long time No. 2 was in the desiccator the triammonic phosphate, which was still present, was partially decomposed.

The loss at 100° shows that at and above this temperature partial dissociation and loss of ammonia takes place; for the loss of $7\text{H}_2\text{O}$ would only give 45'17 per cent.

When left to the temperature of the laboratory in a loosely stoppered flask the product effloresced and lost water slowly. The loss amounted to 6'28 per cent in six weeks.

Dissociation by Water.

Experiment 1.—One gram. of the material was ground up with 100 c.c. of water, and left to digest for three-quarters of an hour. A slightly ammoniacal reaction was observed in the atmosphere of the flask, though it was only detected with difficulty. The solution was filtered with a view to titrating the phosphoric acid separately in the solution and in the precipitate. This latter had become amorphous, but there was always a trace of crystalline material left.

Experiment 2.—This was conducted in the same manner except that the mixture was boiled for three-quarters of an hour. The evolution of ammonia was distinct, and the precipitate entirely amorphous. The solution was filtered, and the solution and precipitate were analysed separately. The following are the results:—

	I.	II.
Phosphoric acid, { In solution ..	7'99	9'62
per cent. . . { Insoluble ..	18'07	16'44
	26'06	26'06

We see that the product is dissociated by cold water, and if we remember that in No. 1 there was still a little of the crystalline material left, we must admit that the decomposition under these conditions is fairly rapid without being instantaneous, and gives both tricalcic phosphate and triammonic phosphate.

When heated, secondary reactions take place and complicate the results.

Although ammonia is given off, very little it is true, more phosphoric acid goes into solution than there should by simple decomposition. There is a double tendency; that of the ammonia to form biammonic phosphate, and that of the lime to become transformed in the alkaline solution into products containing more lime than the tricalcic phosphate.

In no case and at no moment is the least trace of lime found in solution; this, however, was easy to foresee. If the ammonio-calcic phosphate is soluble it is virtually in this sense that the decomposition is instantaneous.

Practically it is only retarded by cohesion. It is thus proved that an ammonio-calcic phosphate really exists, only differing from the ammonio-magnesian phosphate by the amount of water of crystallisation.

I have not yet succeeded in obtaining the ammonio-barytic phosphate by the same method; in dilute solution nothing is obtained, and if the solution is concentrated it is the triammonic phosphate that is first separated.

The ammonio-calcic phosphate being difficult to obtain, as has been shown, it suffices for the solubility or the dissociation of the corresponding baryta compound to be a little greater for the exact conditions of dilution and temperature to be still more difficult to fulfil.—*Bull. Soc. Chim.*, Series 3, vol. xxvii, No. 5.

CHLOROBROMIDES OF THALLIUM. COMPOUNDS OF THE TYPE Tl_4X_6 .

By V. THOMAS.

A FAIRLY large number of chlorobromides belonging to this series have been described. They were prepared by means of either one of the following methods:—

1. *The Action of Bromine on Thallous Chloride in the presence of Water.*—MM. Carl Wiegand and Jos. Meyer (Wiegand, Inaug. Dissert., Berlin, 1899; Meyer, *Zeit. für Anorg. Ch.*, 1900, p. 321) obtained a chlorobromide with the formula $\text{Tl}_6\text{Cl}_5\text{Br}_2$, which they regarded as a mixture of TlCl with a new chlorobromide, $\text{Tl}_4\text{Cl}_4\text{Br}_2$.

Mr. Cushman (*Am. Chem. Journ.*, 1900, p. 222) attributed to the body thus formed the formula $\text{Tl}_4\text{Cl}_4\text{Br}_2$.

2. *The Action of the Chloride on Thallous Bromide in the presence of Water.*—MM. Carl Wiegand and Jos. Meyer obtained a compound, $\text{Tl}_4\text{Cl}_2\text{Br}_4$, decomposed by water into a chlorobromide with the formula $\text{Tl}_4\text{Cl}_3\text{Br}_3$.

3. *The Action of TlCl on TlBr_3 in Solution.*—MM. Carl Wiegand and Jos. Meyer obtained $\text{Tl}_4\text{Br}_4\text{Cl}_2$; Mr. Cushman obtained $\text{Tl}_4\text{Cl}_3\text{Br}_3$.

4. *The Action of TlBr on TlCl_3 in Solution.*—According to the German chemists $\text{Tl}_4\text{Cl}_4\text{Br}_2$ is formed, but according to Mr. Cushman, $\text{Tl}_4\text{Cl}_3\text{Br}_3$.

5. *The Decomposition of the Chlorobromides of the Type Tl_4X_6 on contact with Water.*—The chlorobromide, $\text{Tl}_3\text{Cl}_2\text{Br}_4$, obtained by MM. Carl Wiegand and Jos. Meyer, decomposes on contact with boiling water, giving rise to the compound $\text{Tl}_4\text{Cl}_4\text{Br}_2$. The chlorobromide, $\text{Tl}_3\text{Cl}_2\text{Br}_4$, should decompose in a similar manner.

According to these researches it would appear that there exist three chlorobromides of the type Tl_4X_6 , corresponding respectively to the formulae, $\text{Tl}_4\text{Cl}_4\text{Br}_2$, $\text{Tl}_4\text{Cl}_2\text{Br}_4$, and $\text{Tl}_4\text{Cl}_3\text{Br}_3$.

* According to this writer the compound obtained should be isomeric with that formed from $\text{TlCl} + \text{TlBr}_3$.

In the course of my researches I have always obtained a well-defined chlorobromide, $Tl_4Cl_3Br_3$. This in certain cases was mixed with a very small quantity of a chlorobromide having the formula $Tl_4Cl_4Br_2$. These researches were directed principally to the examination of the raw product resulting from the action of bromine on thallous chloride.

The action of bromine in small quantities on thallous chloride in the cold and in contact with water gives a yellow powder of which the composition is not constant, and depends on the quantity of bromine added. Its composition appears to oscillate between the limits $TlCl$ at $Tl_4Cl_3Br_3$.

The raw product dissolved in water crystallises in flakes mixed with a certain quantity of needles, and the first deposits are always of the same composition, viz., $Tl_4Cl_3Br_3$. It is exactly the same if we add bromine to a warm concentrated solution of thallous chloride. The flakes are produced immediately, or on cooling, and correspond to the formula $Tl_4Cl_3Br_3$.

In the first series of experiments the following method of working was adopted:—9.6 grms. of thallous chloride were rubbed up with a little water (50 c.c. or 100 c.c. for example), then 2 grms. of bromine were added; that is to say, a quantity slightly superior to the amount theoretically necessary to transform the $TlCl$ into a compound of the type Tl_4X_6 (the theoretical quantity is 1.6 grms.).

In this manner, when all the bromine had disappeared, we obtained a yellow powder insoluble in the cold. A large quantity of water was added to make the total bulk about a litre; this was then boiled until everything soluble had gone into solution.

During the heating a slight hydrolysis was observed. The solution was filtered so as to have a perfectly clear liquid, and was then submitted to fractional crystallisations.

On submitting the solution to a very slow cooling we observed at about 40° a deposit of small hexagonal flakes. When the temperature reached 33° the product formed was collected, and its weight was almost 2 grms. (product No. 1). Between 33° and 24° (the temperature of the laboratory in which the experiments were carried out) we obtained a fresh deposit exactly similar in appearance to the first one and of about the same weight (product No. 2).

The solution was then concentrated, and its concentration was such that on cooling about 3 grms. of a product were obtained, distinctly different from the others in that it was not homogeneous (product No. 3). It consisted to a great extent of very well formed needles, often grouped together in fern-like clusters. These needles were mixed with hexagonal flakes analogous to those of the preceding deposits.

Further, all these products have a more or less deep orange colour; the needles have the greatest depth of colour.

If we continue to concentrate the mother-liquor we obtain hexagonal flakes (product No. 4); then when the concentration is pushed further we obtain a fresh crop of needles.

Up to the present we have not succeeded in preparing these needles directly in a state of absolute purity.

The conditions under which they can be prepared, independently of the hexagonal flakes, are not yet known.

Two facts only ought to be considered as being well proved; that is to say, the influence exercised on the formation of this body by the concentration of the solution on the one hand, and by the rate of cooling on the other.

To observe its formation it appeared to be necessary to operate on concentrated solutions and to avoid rapid cooling.

The following results of analysis are those of the different products obtained from the same preparation. The estimations of the total halogens and the separation of the chlorine and bromine were made on different portions:—

	AgCl+nAgBr. Cl.	Br.	Tl
			(thallous).
<i>Product No. 1.</i>			
Homogeneous. Orange coloured hexagonal plates	84.51 84.13	8.96	20.58 52.35
<i>Product No. 2.</i>			
Homogeneous. Orange coloured hexagonal plates	84.51 85.03	8.75	20.90 52.53
<i>Product No. 3.</i>			
Needles mixed with hexagonal plates ..	84.92 83.13 84.46	10.79	17.34 —
<i>Product No. 4.</i>			
Orange yellow hexagonal plates	84.20	11.52	16.15 —

Whatever the preparations may be these figures are constant. The analysis of compounds corresponding to those in the first two horizontal lines, but coming from different preparations, gave $AgCl+nAgBr$, 86.91 and 84.93, 84.46 and 84.85, 85.30 and 84.84; Cl, 8.94, 8.84, and 9.13; Br, 20.44, 20.53, and 20.70.

For the non-homogeneous products mentioned in the two last lines the figures found by analysis were:— $AgCl+nAgBr$, 84.60, 84.13, and 84.53; Cl, 10.40 and 10.07; Br, 17.77 and 18.88.

These analytical results are all very interesting. They show distinctly that under the conditions of the experiment:—

1. The first deposits have always the same composition, and correspond exactly with the formula $Tl_4Cl_3Br_3$, which requires $AgCl+nAgBr$, 85.54; Cl, 9.15; Br, 20.65.

2. The proportion of bromine is lowered, and that of the chlorine increased at the same time that the needles appear.

3. The deposits which form after the appearance of the first needles, even when they consist entirely of hexagonal plates, are distinguished from the preceding deposits by a smaller proportion of bromine and a higher proportion of chlorine.

Under these conditions there still remains a very important point to elucidate. Have the needles which are formed at the same time as the plates the same composition as these latter, or do they correspond to a different atomic ratio between the thallium, the chlorine, and the bromine?

With a view to solving this problem we felt constrained to operate on a much greater quantity of thallous chloride as the original material, and to separate out the needles formed one by one.

For this purpose we took the following weights:— $TlCl$, 36 grms.; Br, 3 c.c.; water 2 litres.

The deposit obtained was treated with boiling water, and the solution left to itself in a suitable place to avoid any rapid fall of temperature. The crystals deposited were picked out and collected one by one with the help of a low power lens; then, as the needles often had small hexagonal plates attached to their sides, they were again gone over, and picked out with the help of a stronger lens, or even, if necessary, a microscope. In a week a sufficient quantity was collected to enable several analyses to be made.

These left no doubt as to the nature of the product, which corresponded to the formula $Tl_4Cl_3Br_3$.

Analysis.—Found, $AgCl+nAgBr$, 84.65 and 85.96; Cl, 13.32; Br, 14.53. Calculated for $Tl_4Cl_3Br_3$: $AgCl+nAgBr$, 84.97; Cl, 12.70; Br, 14.31.

The plates and the needles* appeared to belong to the

* In a note published in the *Comptes Rendus*, November 4, 1901, we gave this body the formula $Tl_4Cl_3Br_3$, through a mistake which occurred in calculating the formula $Tl_4Cl_3Br_3$ and $Tl_4Cl_4Br_2$; we took this opportunity of making this correction as our analyses were not published. This formula agrees very well with the note which appeared in the *Comptes Rendus*, of November 26, 1900. They are contradictory with regard to those in my paper of January 14, 1901.

same crystalline system, but we could not be certain on this point without obtaining measurable crystals, and making the usual measurements.

We can pass, however, very easily from plates to needles, and from needles back to plates. The experiment can be repeated indefinitely by taking a hot saturated solution either at a constant or a variable temperature.

This phenomenon shows us distinctly how complex are the conditions of equilibrium which affect the formation of such and such a chlorobromide.

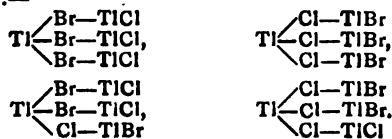
It is especially the chlorobromide, $Tl_4Cl_3Br_2$, which is formed by the decomposition of the $Tl_3Cl_2Br_4$, and obtaining the needles $Tl_4Cl_3Br_2$ is extremely interesting, as this body would represent the hypothetical chlorobromide which Wiegand prepared mixed with $TlCl$, the mixture having the rough formula $Tl_3Cl_2Br_4$. It should also be identical with the plates $Tl_4Cl_3Br_2$ prepared by Cushman.

Both needles and plates have exactly the same properties. Under the action of heat, either when heated directly or when heated in the solution from which they are deposited, they undergo a remarkable change of colour. When hot they take a blood-red colour; this phenomenon is, in fact, produced with a very slight elevation of temperature.

Towards 35° the original colour changes in a very distinct manner. This red colouration disappears more or less rapidly on cooling, and gives place to the original orange colour. When heated in Maquenne's apparatus the two chlorobromides attack the copper and fuse at about 240° .

The chlorobromides of the type Tl_4X_5 are also of great interest from the theoretical point of view. Werner's theory, like that of Blomstrand-Jørgensen on the constitution of double salts, enables us to predict two isomeric derivatives for such compounds.

We might have, for example, according to the Blomstrand-Jørgensen theory, the following compounds as isomers:—



By Werner's hypothesis we ought to admit an atom of Tl placed in the centre of an octahedron, and the two salts in question would become stereo-isomers according to the position of the atoms TlX_6 .

According to Cushman, these two theoretical stereo-isomers would correspond in practice to different substances. This writer claims to have obtained, under the following circumstances, the isomers of the formula $Tl_4Cl_3Br_2$:—

When thallic bromide, in suspension in warm water, is treated with a dilute solution of thallic chloride an orange-coloured compound is obtained; on dissolving, by boiling the solution and then re-cooling, orange-coloured hexagonal plates are deposited. For the sake of brevity we will call this compound α .

On the other hand, if thallic chloride is suspended in a considerable quantity of warm water and treated with a warm concentrated solution of thallic bromide, a blood-red compound is produced which, on re-dissolving by heat and then rapidly re-cooling the solution, gives a deposit of blood-red hexagonal crystals; we will call this product β .

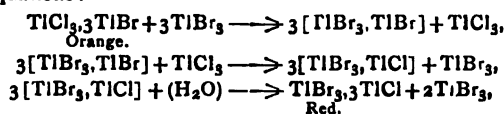
Now, according to Mr. Cushman, the derivatives α and β are isomeric. The derivative α is stable; the derivative β , on the other hand, seems to be characterised by its instability.

We have not repeated these experiments, as the details for carrying them out are entirely wanting.

In any case it is probable that the existence of a few needles in the plates submitted to analysis does not exercise any great influence on the proportion of chlorine; in fact, for $Tl_4Cl_3Br_2$ we ought to have Cl , 9.15; and for $Tl_4Cl_3Br_2$, Cl , 12.70.

At the same time we were surprised that the facts observed by us had not been noticed as much as those published by MM. Wiegand and Jos. Meyer and Mr. Cushman. In no paper have we found any mention of needles of the composition $Tl_4Cl_3Br_2$, and in no paper is the change of colour which is so characteristic of the compounds of the type Tl_4X_5 mentioned.

We cannot, however, admit that the so-called stereo-isomers of Mr. Cushman are identical with the compounds $Tl_4Cl_3Br_2$ (orange and red modifications) that we have previously described; all we have been able to find in his work seems, on the contrary, to show a deep division between the two series of bodies. The derivative δ (red derivative) appears only to be obtained in the presence of an excess of thallic bromide, and the derivative α in the presence of an excess of thallic chloride. We could easily pass from one form to the other according to the equations:—



This question of isomerism should be proceeded with cautiously, and we have no doubt but that Mr. Cushman while continuing his researches will eventually be able to throw greater light on the results of his earlier work.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 11.

THE PRECIPITATION AND SEPARATION OF SILVER IN THE ELECTROLYTIC WAY.*

By W. H. FULWEILER and EDGAR F. SMITH.

THE fact that silver can be quantitatively determined in the electrolytic way is well known. It has had ample confirmation in this laboratory, where numerous estimations of the metal in cyanide solution have been made. It is probable, too, that it was here that the first quantitative results with this electrolyte were obtained, although it is customary to credit the method to Luckow, notwithstanding, in his published account of the electro-deposition of silver from its double cyanide solution, he presents no quantitative data, and merely remarks in regard to such solutions:—"So fällt aus solchen Lösungen das Silber gleichmässig in regulinischer form mit mattem Metallglanze nieder" (*Zeit. Anal. Chem.*, xix., 15). But, be that as it may, without any further reference to the origin of the method, the latter is most deserving of consideration, and to extend in some measure its general applicability we have made separations of silver from solutions in which three or four other metals were simultaneously present. It is the results in this direction which we most desire to put upon record.

Those interested in electro-chemistry will at once recall that in the hundreds of separations made with the current more than two metals rarely were present in the electrolyte. Now that electro-chemical analysis is daily becoming more prominent, the proper working conditions for such problems should be ascertained. It is probable that this phase in electrolysis has been too long neglected. We give first results obtained with silver alone (Table I.).

In trials 1 and 2 the metal was precipitated upon a dish, while in 3 and 4 a plate cathode, and in 5 and 6 a cone was used to receive the silver, which was very adherent and brilliant in lustre. It was washed with water, alcohol, and ether.

Silver from Copper.

The first thought was to further test the separation of silver from copper, making a special point in varying the quantity of the latter metal (Table II., A).

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xxiii., No. 8.

TABLE I.

	Silver. Grm.	Dilution. C.c.	Potassium cyanide. Grms.	Current. N.D. ₁₀₀	Volts.	Temperature.	Time. Hours.	Silver found. Grm.
1	0.2133	125	2	0.03 A	2.5	65°	4	0.2132
2	0.2133	125	2	0.03 A	2.5	60°	3	0.2133
3	0.2133	125	4	0.04 A	2.5	60°	3	0.2131
4	0.2133	125	2	0.025 A	2.7	60°	4	0.2134
5	0.2133	125	2	0.025 A	2.7	60°	3	0.2135
6	0.2133	125	2	0.025 A	2.7	60°	4	0.2125

TABLE II.—Silver from Copper.

Silver. Grm.	Copper. Grm.	Potassium cyanide. Grms.	Dilution. C.c.	Current. N.D. ₁₀₀	Volts.	Temperature.	Time. Hours.	Silver found. Grm.
A.								
0.1066	0.1053	2	125	0.021 A	1.2	60°	4½	0.1066
0.1066	0.1053	2	125	0.02 A	1.25	55°	6	0.1064
0.1066	0.1053	2	125	0.02 A	1.25	55°	6	0.1065
B.								
0.1066	0.2106	2	125	0.02 A	1.25	60°	3½	0.1066
0.1066	0.2106	2	125	0.02 A	1.25	65°	3½	0.1065
C.								
0.1066	0.5265	4	125	0.03 A	1.2	65°	3½	0.1066
0.1066	0.5265	4	125	0.02 A	1.2	60°	3½	0.1068

TABLE III.—Silver from Copper and Cadmium.

Silver present. Grm.	Copper present. Grm.	Cadmium present. Grm.	Potassium cyanide. Grms.	Dilution. C.c.	Current. N.D. ₁₀₀	Volts.	Temperature.	Time. Hours.	Silver found. Grm.	
A.										
0.1066	0.1053	0.1128	3	125	0.02 A	1.25	65°	3½	0.1063	
0.1066	0.1053	0.1128	3	125	0.015 A	1.25	65°	4	0.1069	
0.1090	0.1053	0.1128	3	125	0.02 A	1.25	65°	4	0.1090	
B.										
Zinc present. Grm.										
0.1090	0.1053	0.1128	0.1244	4	125	0.02 A	1.25	80°	5	0.1092
0.1090	0.0526	0.0564	0.0622	5	125	0.02 A	1.25	75°	6	0.1089
0.1090	0.0526	0.0564	0.0622	3.5	125	0.02 A	1.25	75°	5	0.1087

TABLE IV.—Silver from Copper, Cadmium, Zinc, and Nickel.

Silver. Grm.	Copper. Grm.	Cadmium. Grm.	Zinc. Grm.	Nickel. Grm.	Potassium cyanide. Grms.	Dilution. C.c.	Current. N.D. ₁₀₀	Volts.	Temper- ature.	Time. Hours.	Silver found. Grm.
0.1090	0.0526	0.0564	0.0622	0.0411	4.5	125	0.02 A	1.2	75°	5½	0.1086
0.1090	0.0526	0.0564	0.0622	0.0411	3.5	125	0.02 A	1.2	75°	5	0.1085

The silver was completely precipitated. It was found to be free from copper.

The next ratio was 1Ag:2Cu (Table II., B).

With the ratio 1Ag:5Cu the results were as given in Table II., C.

The experience gathered from these determinations shows that the separation of these two metals can be conducted in this particular way with the certainty of success. In several instances the liquid from the silver was diluted to 500 c.c. and electrolysed with a more powerful current when the copper was fully precipitated, and showed the absence of silver upon applying the customary tests.

Silver from Copper and Cadmium.

(See Table III., A).

The next step was the introduction of a zinc salt into the electrolyte. Strangely enough some cadmium was now found in the deposit of silver, but it was not long before the observation was made that if the electrolyte was heated to 75–80° before passing the current the co-precipitation of cadmium could be absolutely prevented. The conditions are indicated in Table III., B.

The silver was found free from any of the other metals. In all the experiments made with the conditions as re-

corded in the preceding paragraphs, the striking brilliant lustre of the deposited silver was particularly marked.

Silver from Copper, Cadmium, Zinc, and Nickel.

(See Table IV.).

None of the other metals were found in the silver deposit. The addition of the nickel to the solution seems to retard the precipitation of silver to a slight degree.

A Method for Detecting Free Phosphorus.—P. Muckerji.—The material in which it is sought to detect free phosphorus is placed in a hydrogen generator ($\text{SO}_4\text{H}_2 + \text{Zn}$), giving an abundant flow of gas at a temperature of 60–70°; the apparatus is placed in the dark, and we notice whether the unlighted gas emits any light. This method is at least as sensitive as that of Mitscherlich. Phosphites and hypophosphites, and phosphides of hydrogen do not give any phosphorescence. The substances which interfere with the phosphorescence of phosphorus in the air under ordinary conditions have their influence diminished in this case. When the operation is finished it is necessary to displace the gas by filling the apparatus with water to prevent spontaneous combustion.—*Zeit. Anorg. Ch.*, vol. xxvii., p. 72.

NOTICES OF BOOKS.

Twenty-sixth Annual Report of His Majesty's Inspectors of Explosives; being their Annual Report for the Year 1901. London: Eyre and Spottiswoode. Edinburgh: Oliver and Boyd. Dublin: E. Ponsonby. 1902. Pp. 230.

THE only modifications of the law made during the past year have been two Orders of Secretary of State of limited application, which will be found in Appendices F (1) and F (2). The former of these deals with the packing of electric detonators, and the latter grants an exemption for acetylene, when compressed into certain porous substances, from being a prohibited explosive.

The growth of trade in explosives has continued during the early part of the past year, but there have been some signs that during the latter half of the year the rate of increase was not sustained.

We are glad to note that as a result of the inspectors' visits to the various factories and magazines throughout the kingdom there is no reason to complain that there is any falling off in their condition or management. Occasions when such serious irregularities are found as to necessitate legal proceedings are now rare. The number of deaths (ten) from accidents by fire or explosion in the manufacture of explosives is unfortunately above the average for the decade (5.4), but this number of deaths is not excessive, and will compare favourably with the death-rate in other dangerous trades.

The total number of factories under continuing certificate or license is 150. This does not include "small firework" or "toy firework" factories. Of the above total number, four factories are under notice of temporary disuse. The total number of factories which have come into existence since the commencement of the Act has been 150, but 55 have become extinct, so the net increase is 95; for the present year there is no increase.

There have been twenty articles added to the authorised list of explosives, and two removed.

During the year, 249 visits have been paid to factories, but in very few cases had proceedings to be taken.

The number of accidents in factories is 76, causing 10 deaths and injuring 38 persons. These accidents are dealt with in detail, but a considerable proportion of them are of an unimportant character, such as the fuming off of gun-cotton in the acid centrifugals, while some of the injuries were only slight.

There are now 393 magazines under continuing certificate and license, being an increase of 8 on the number for 1900, and these have been visited 462 times; in very few instances were serious defects or irregularities found, and, in general, the condition of magazines throughout the country is highly satisfactory. No legal proceedings have been taken throughout the year.

The amount of foreign nitro-glycerin compounds imported in 1901 is 1,473,950 lbs., against 998,030 lbs. in 1900; there has also been an increase in the importation of blasting explosives not containing nitro-glycerin from 129,750 lbs. in 1900 to 142,900 in 1901.

With regard to the chemical work of the department, conducted by Dr. Dupré, F.R.S., 427 articles have been examined; this is a slight decrease as compared with the previous year. These articles are divided into three classes:—

1. Samples of licensed explosives and materials used in the manufacture of such explosives.

2. Articles which do not properly come under the above heads.

3. All explosives examined in connection with the Woolwich testing station.

Of the first class, 335 were passed and 44 rejected; the somewhat larger number under the heading of "rejected" is mainly due to the fact that owing to requirements under the Mines Act, the compositions of explosives permitted to be used are fixed more closely than in former years. Much

work has been done in order to keep analytical methods fairly abreast of requirements. The compositions of explosives generally are becoming more complicated, and the limits within which variations are allowed are being drawn more and more narrowly; the correct analysis of explosives thus becomes increasingly difficult. Some explosives contain as many as eight constituents, and their correct analysis is often a matter of some difficulty.

A number of accidents and explosions abroad are described and commented upon, and the report closes with the usual appendices giving full details of the various matters referred to in the body of the report.

La Grande Industrie Chimique Minérale. ("The Industries of Mineral Chemistry"). By E. SOREL. Paris: C. Naud. 1902.

THIS treatise differs in some respects from the ordinary run of books on industrial chemistry. In many instances it must be supplemented by reference to books on general chemistry for details concerning the characteristic properties of the substances considered. On the other hand, the author's wide experience eminently fits him to produce a comprehensive and complete work of reference for the use of the manufacturer. Nearly half the volume is occupied by the study of sulphur and its derivatives, the manufacture of sulphuric acid naturally receiving special attention. This part is well balanced and exact, and should commend itself to the student of chemical industries. Under nitrogen are included nitrates and nitric acid, ammoniacal salts, and compounds of cyanogen. The third part is devoted to the phosphates, treated both from an agricultural and industrial point of view. Here the treatment is occasionally not altogether satisfactory, as, for instance, in the case of the phosphoric acids; and some omissions may be noticed. In fact, the end of the book hardly reaches the same standard of excellence as the beginning, and signs of less careful writing may be detected. The last part includes the alums and vitriols of iron, copper, and zinc. The usefulness of the book is seriously impaired by the absence of an alphabetical index.

Rapports du Jury International de l'Exposition Universelle de 1900. Classe 90. Parfumerie. ("Reports of the International Jury of the Paris Exhibition. Class 90. Perfumery"). By L. V. PIVET. Paris: Imprimerie Nationale. 1901.

THIS report of the international jury on the perfumery class of the Paris Exhibition forms a concise and useful account of the subject. The historical chapters include tables illustrating the state of the French export trade in essences and perfumes generally. The second of the five parts into which the book is divided treats of the rôle played by science in the industries; in an introductory chapter on the constitution and physiology of odiferous bodies, theoretical considerations are shortly reviewed; the chapter on the chemical constituents of essences gives physical and other data concerning various substances of importance in perfumery; unfortunately, however, the information is not always accurate; for instance, a CH group is omitted in the formula of cinnamic aldehyde, which is written $C_6H_5-CH=COH$, and the boiling-point of the same substance is incorrectly given. The methods of analysis of the natural products, essential oils, and essences, and the state of the industry at the present time in various countries, occupy the third and fourth parts; the latter includes tables of exportation from, and importation into, France for the year 1900, and the volume ends with a list of awards in this and previous Exhibitions. The study of the subject of perfumery is one which requires a considerable knowledge of chemistry, and the employment of methods based on scientific foundations should tend to increase and develop the trade; this book, quite

apart from its reference to the Exhibition, appears to be a useful little handbook and compilation.

Beiträge zur Chemischen Physiologie und Pathologie. ("Contributions to Chemical Physiology and Pathology"). Edited by FRANZ HOFMEISTER. Band II. 5 and 6 Hft. Braunschweig: Friedrich Viewig und Sohn. 1902.

THE May number of this journal opens with a paper by Dr. Paul Mayer, describing his researches on the formation of indoxyl, phenol, and glycuronic acid, in cases of phloridzin diabetes; the author believes that he has established the fact that no increase takes place in the formation of any of these substances, a conclusion which does not agree with Lewin's results published some months ago in an earlier number of the journal. C. Neuberg and F. Blumenthal contribute a paper on the formation of iso-valeric acid and acetone by the oxidation of gelatin; and much interest attaches to a communication by Dr. P. Bielsfeld on the subject of the iron contained in human liver cells. The question of the origin of the various compounds of iron known to occur in the liver is not discussed, but the results of many careful and exact determinations of the iron in normal liver cells are given, with a view to determining in which sex and at what age, generally speaking, maxima of percentages of iron are reached. The systematic examination of these and similar details should help to add to our at present scanty knowledge concerning the connection between the simple and complex iron salts found in the human liver.

CORRESPONDENCE.

THE MELTING-POINT OF CHROMIUM.

To the Editor of the Chemical News.

SIR,—In my recent note on "The Melting-point of Chromium" (*CHEMICAL NEWS*, vol. lxxvi., p. 13), I stated that the chromium contained 99 per cent of chromium; this was the approximate amount the makers said it contained. An analysis of it gave—

Aluminium	traces
Iron	1.23 per cent
Silicon	0.14 "
Chromium	98.63 "
	100.00 "

Mr. Hogg, in his letter (p. 35), says that aluminium is "reputed to lower the melting-point of certain metals enormously." This is a mistake, as small percentages of aluminium do not lower the melting-points of metals to the extent it was at one time supposed. In the "Third Report to the Alloys Research Committee of the Institution of Mechanical Engineers," Roberts-Austen showed that the addition of 1 per cent of aluminium to pure iron did not lower the melting-point much below the melting-point of pure iron; and I do not see why, even if it was present to the extent of 1 per cent, it should lower the melting-point of chromium.

In a recent paper (see *Journal of the Society of Chemical Industry*, June 30, 1902) I pointed out that the melting-point of manganese, usually stated to be 1900° C., was only 1280° C., and I thought it would be interesting to determine if chromium had the high melting-point usually attributed to it, namely, considerably over 1775° C.

Mr. Hogg quotes some experiments by Osmond on the melting-point of steel; but it is the carbon which is most probably the cause of the lowering of the melting-point of steel.

I do not claim that my determinations of the melting-point of chromium and manganese are absolutely correct, but I think they will be found to be within 30° C. of the melting-point of the pure metals when they are obtained

chemically pure in sufficient quantity to enable accurate determinations to be made.—I am, &c.,

ERNEST A. LEWIS.

310, Dudley Road, Birmingham,
July 23, 1902.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Bulletin de la Société Chimique de Paris.

Series 3, Vol. xxvii., No. 1.

Alloys of Aluminium and Magnesium. — O. Boudouard.—Already noticed.

Action of Fuming Sulphuric Acid on Ethanal, Propanal, and Propanone.—Marcel Delépine.—Trioxymethylene has been shown to react on fuming sulphuric acid, giving a kind of sulphuric acetal of methanal. Similar experiments made with ethanal and propanal have given results of an entirely different character. These aldehydes are sulphurised; the same is the case with acetone. The author then gives the details of a large number of experiments and the analysis of the products obtained.

Synthesis of Dimethylsuccinic Acid under the Influence of Light.—W. Sernow.—If iodopropionic ether is exposed to direct light in the presence of metallic mercury, we observe that the mercury is gradually changed into a reddish crystalline powder of mercuric iodide, and finally the ether contains no more iodine. By shaking up, by means of a motor, 20 grms. of the ethylic ether of α -iodopropionic acid in alcoholic solution with 30 grms. of mercury in a sealed flask in direct sunlight, the reaction is complete in a few sunny days. In this manner the author obtained a product which, when separated with alcohol, took the form of a thick oil. It commences to boil at about 220°, and at 230° the greater part distils over; at 300° and over, decomposition takes place. The 220–230° portion, after several redistillations, boils at 219–220°; its formula is $C_{10}H_{18}O_4$, thus dimethylised succinic ether is formed.

No. 2.

The presence of Tellurium in Ingots of American Silver.—C. Vincent.—Already inserted in full.

Nitromannite and Nitrocellulose.—L. Vignon and F. Gerin.—Already noticed.

Reducing Properties of certain Nitric Ethers.—L. Vignon and F. Gerin.—Already noticed.

Nitric Derivatives of Pentaerythrite.—L. Vignon and F. Gerin.—Already noticed.

Nitrated Derivatives of Arabite and Rhamnite. Constitution of certain Nitric Ethers.—L. Vignon and F. Gerin.—Already noticed.

The Microchemical Detection of Potassium, Rubidium, Cæsium, Indium, and the Hyposulphites.—A. C. Huysse.—The well known precipitates formed by the alkaline salts in the presence of hyposulphite of sodium and bismuth have very characteristic microcrystalline forms. The reagent is prepared by dissolving a little sub-nitrate of bismuth in a watch-glass, with the least possible quantity of hydrochloric acid, then adding water until a permanent precipitate is formed, which is re-dissolved in hyposulphite of soda. This solution is treated with alcohol until it commences to appear cloudy, and is clarified by adding a drop of water. The advantage of this method is that ammoniacal salts give no precipitate with the reagent. Hyposulphite of sodium, on the other hand, forms with thallous nitrate a microcrystalline precipitate, which may be used in the absence of the halogen salts.—*Zeit. Anal. Ch.*, vol. xxxix., p. 9.

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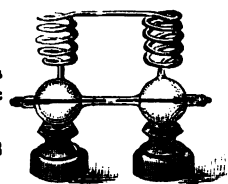
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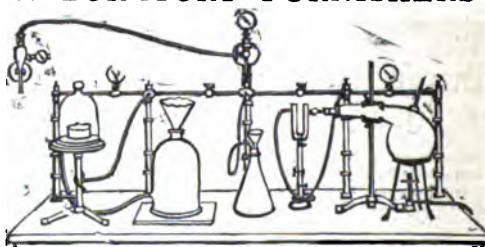
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THE CHEMICAL NEWS.

VOL. LXXXVI., No. 2228.

ON THE ATOMIC WEIGHT OF RADIUM.

By M^{me}. CURIE.

By concentrating by fractional crystallisation the greater part of the radiferous barium at my disposal I have succeeded in obtaining about 1 decigram. of perfectly pure radium chloride. This has enabled me to determine the atomic weight of radium.

It results from the following experiments that the atomic weight of radium is 225 (taking Cl = 35.4 and Ag = 107.8) with a probable uncertainty of not more than one unit, radium being considered a bivalent element.

The method employed consists in estimating, in the state of chloride of silver, the chlorine contained in a known weight of anhydrous radium chloride. As a control experiment I determined the atomic weight of barium by the same method, under the same conditions, and with the same quantity of material. The numbers found always fell between 137 and 138. I have also found that this method gives satisfactory results even with a very small quantity of material.

Many determinations were made with radium chloride; after each operation the radium was brought to the state of chloride in the following manner:—The liquid, containing after the estimation radium nitrate and silver nitrate in excess, was mixed with pure hydrochloric acid. The silver chloride was separated by filtration, and the filtrate was evaporated to dryness several times with an excess of pure hydrochloric acid. Experience shows that all the nitric acid may be eliminated in this way.

The weighings were performed on an aperiodic Curie balance in perfect adjustment, and weighing to the twentieth of a m.grm. This direct reading balance permits of very rapid weighings, an essential condition for weighing the anhydrous chlorides of barium and radium, which slowly absorb water in spite of the presence of desiccating agents in the balance. The substance to be weighed is placed in a platinum crucible; this crucible has been in use a long time, and I have verified that its weight does not vary a tenth of a m.grm. in the course of an operation.

The hydrated chloride obtained by crystallisation was heated in an oven to transform it into the anhydrous chloride. Experience shows that when the chloride was kept for some hours at 100° its weight does not alter, even when the temperature rises to 200° and is kept there for some hours. Therefore the anhydrous chloride so obtained constitutes a perfectly definite body. In all the estimations the chloride was dried at 150°.

M. Demarcay has been good enough to examine the spectrum of the radium chloride submitted to analysis, and has given me valuable indications of the degree of purity of the body.

Two series of experiments were made. The first was with a radium chloride which M. Demarcay considered sensibly pure, but in which the spectrum showed still the three principal lines of barium with good intensity. The numbers obtained in four successive operations were the following:—

220.7 223.0 228.8 223.1

I then undertook a new purification, and succeeded in obtaining a product much more pure. M. Demarcay considers that this second product only contains a minimum quantity of barium incapable of influencing appreciably the atomic weight.

Here are the results of three determinations made with this perfectly pure radium:—

225.3 225.8 224.0

The mean of these numbers is 225.0. I think this number is within one unit of the truth.

The silver chloride from the estimations was always radio-active and luminous. I satisfied myself that it had not carried down a ponderable quantity of radium. I have found also that the weight of radium chloride regenerated did not vary in the operations.

The separation of the radium chloride was effected by fractionally crystallising in a hydrochloric solution a radiferous barium chloride which already had been purified carefully. When the concentration of radium becomes of a certain strength the crystals, at first colourless in the mother-liquor, become yellow or rose coloured a few hours after their deposition. This colouration disappears on solution. It appears to be due to the simultaneous presence of barium and radium, for crystals of pure radium chloride do not become coloured. This observation may be useful for following the course of the fractionation.

Pure anhydrous radium chloride is spontaneously luminous.

From its chemical properties radium is an element of the alkaline-earthly series, and in this series it is the higher homologue of barium. According to its atomic weight it should be placed in Mendeleeff's table below barium in the alkaline-earthly series, and on the line with thorium and uranium.—*Comptes Rendus*, cxxxv., p. 161, July 21, 1902.

ON THE MEASUREMENT OF TEMPERATURE.*

PART I.—ON THE PRESSURE COEFFICIENTS OF HYDROGEN AND HELIUM AT CONSTANT VOLUME AND AT DIFFERENT INITIAL PRESSURES.

PART II.—ON THE VAPOUR PRESSURES OF LIQUID OXYGEN AT TEMPERATURES BELOW ITS BOILING-POINT ON THE CONSTANT VOLUME HYDROGEN AND HELIUM SCALES.

PART III.—ON THE VAPOUR PRESSURES OF LIQUID HYDROGEN AT TEMPERATURES BELOW ITS BOILING-POINT ON THE CONSTANT VOLUME HYDROGEN AND HELIUM SCALES.

By MORRIS W. TRAVERS, D.Sc.,
Fellow of University College, London,
GEORGE SENTER, B.Sc.,
and
ADRIEN JAQUEROD, D.Sc.

Part I.—By M. W. TRAVERS and A. JAQUEROD.

PRESSURE COEFFICIENTS OF HYDROGEN AND HELIUM.

THE pressure coefficients have been determined by measuring the pressures which the gases exert when the bulb of the thermometer is in melting ice, or in steam at the boiling-point. Full details of the method employed are given in the paper of which this is an abstract.

The principal new features of the method are as follows:—The gases were introduced into a glass bulb sealed to a capillary glass stem, which was in turn sealed at the other end to the tube which formed the "dead space"; thus eliminating any chance of leakage of the gas, which must necessarily occur when steel tubes, connected to the glass by cement, are employed. The mercury in the dead space was brought close to, but not into contact with, a point in the dead space, and the pressure on the gas in the thermometer was directly observed by measuring the height of the mercury in an exhausted manometer tube above the mercury in the dead space; the apparatus was so arranged that the two mercury menisci lay on the same vertical axis.

The mercury column and the dead space were enclosed between two parallel glass plates in a water-jacket, the temperature of which could be maintained constant, by means of a rapid current of water, to within 0.02° C. In-

* Abstract of a Paper read before the Royal Society, June 19, 1902.

equalities in the temperature of the mercury column and dead space, to which the largest errors in such measurements are due, were thus eliminated.

The scale which formed the first surface of the water-jacket was certainly correct to 0.01 m.m. The distances between the surfaces of the mercury menisci and the nearest division of the scale were measured by means of a telescope with an ocular micrometer placed at a distance of one metre from the apparatus. By this means it was possible to obtain readings concordant to 0.01 m.m.

In calculating the pressure, corrections were made for capillarity, taking into consideration the height of each meniscus, which was measured at each observation, and for the temperature of the column. The volume of the dead space, which varied with the distance of the mercury in it from the point, and with the height of the meniscus, and the expansion of the bulb with the change of temperature and pressure between 0° and 100° C., were taken into consideration in making the final calculations.

In determining the value of the coefficients, the pressures P_0 and P_{100} which the gas would exert, supposing the whole of it was at the temperature of melting ice or of saturated steam under normal pressure, we first calculated. Each of the values of P_0 or of P_{100} given in the following table, is the result of four consecutive measurements of the pressure, temperature, &c.

Pressure Coefficient of Hydrogen.

Series I. (a)	P_0	604.458, 604.452
	P_{100}	948.789, 948.824, 941.809
	α	0.00366261
(b)	P_0	666.103, 666.102
	P_{100}	951.059, 951.044
	α	0.00366252
(c)	P_0	706.528
	P_{100}	965.291
	α	0.00366246
Series II.	P_0	520.326, 520.311
	P_{100}	710.897, 710.882, 710.907
	α	0.00366268

Pressure Coefficient of Helium.

Series I. (a)	P_0	690.232, 690.238
	P_{100}	743.044, 943.044, 942.992
	α	0.00366241
(b)	P_0	671.422, 671.418
	P_{100}	917.322, 917.328, 917.352
	α	0.00366270
Series II. (a)	P_0	522.984, 522.984
	P_{100}	714.576, 714.529, 714.577
	α	0.00366313
(b)	P_0	523.016, 523.020
	P_{100}	714.568, 714.583
	α	0.00366255

We have thus three determinations of the pressure coefficient of hydrogen at an initial pressure of 700 m.m., to the third of which, since it is the result of one measurement of P_0 and one of P_{100} , and was only carried out to make certain that our instrument was in good working order before determining the pressure coefficient for helium, we attach less weight than to the remaining values. The results of the two experiments with helium are of equal value, each being the result of two determinations of P_0 and three determinations of P_{100} . The mean values of the pressure coefficient for both hydrogen and helium appear to approach 0.00366255, a number which agrees very closely with the value obtained by Chappuis (0.00366254), and is somewhat lower than that given by Onnes (0.0036627) as his final result, though differing little from one of his three actual measurements (0.0036625).

The values of the coefficients at lower initial pressures do not show the same concordance as those at higher pressure, but they tend to show that at very low pressures the pressure coefficient does not assume a lower limiting value, the reciprocal of which should be the melting-point

of ice on the absolute scale of temperature. That helium and hydrogen have the same pressure coefficient, and that the coefficient is independent of the pressure, suggest that whatever correction may be necessary to reduce temperatures between 0° and 100° C. on the scale of the hydrogen or helium constant-volume thermometer to temperatures on the absolute scale, it must be very small. Further study of the thermodynamic properties of these gases is necessary for the solution of this important problem.

Part II.—By M. W. TRAVERS, G. SENTER, and
A. JAQUERON.

VAPOUR PRESSURES OF LIQUID OXYGEN.

As has been shown in Part I., the coefficients of increase of pressure at constant volume for hydrogen and helium, between 0° and 100° C., have the same value, viz., 0.00366255 or 1/273.03.

Numerous measurements of the boiling points and vapour pressures of liquid oxygen on the constant volume hydrogen scale have been made by previous investigators. The values obtained differ by two or three degrees, and even the most reliable measurements vary between -182.4° and -182.7° C. In very few cases are any experimental details given in the original papers; in most cases it is even impossible to ascertain what value was taken for the coefficient of expansion of hydrogen. In every case, however, it appears that the measurements were made by immersing the thermometer in a mass of liquid oxygen, and measuring the pressure under which the liquid was evaporating. As is shown in our paper, it is extremely difficult to maintain liquid oxygen in a steady state of ebullition, and unless a rapid current of air or oxygen is passed into the liquid it ceases to boil, and may become superheated to the extent of more than one degree. Further, it is very difficult to obtain a sufficient quantity of pure oxygen for such a measurement.

In our experiments a bulb in which a small quantity of pure oxygen could be liquefied was immersed together with the thermometer bulb in a vacuum-vessel containing liquid air or oxygen, through which a fairly rapid current of air was passed. The bulb containing the pure oxygen communicated with the lower chamber of a barometer, so that the measurements of the vapour pressure were quite independent of the atmospheric pressure, with a mercury pump, and with an apparatus for generating pure oxygen from potassium permanganate. Simultaneous readings of this barometer and of the thermometer, which contained hydrogen or helium, gave the vapour pressures of pure oxygen at temperatures which could be varied between 80° and 90° Abs., according as the vacuum vessel surrounding the thermometer bulb, &c., contained freshly made liquid air or nearly pure oxygen.

Four thermometers were employed in these measurements, the capacities of the bulbs being approximately 90 c.c., 12 c.c., 26 c.c., and 27 c.c. Only one single set of measurements on the hydrogen scale were made with the large thermometer, which was the same instrument as was employed in the determination of the pressure coefficients of the gases, for though it was possible to observe a steady temperature by means of it with a degree of accuracy approaching one part in 20,000, it was found impossible to maintain so large a thermometer bulb at a constant and uniform temperature, without employing very large masses of liquid oxygen. The results obtained by means of this thermometer differ only by 0.1° from the mean of those obtained by means of the three smaller thermometers. The temperatures observed by means of the three smaller thermometers rarely differ by more than 0.03° from the temperature, corresponding to the same pressures, taken from the smooth vapour pressure curve.

The form of the thermometers employed in this research, and in the measurements of the vapour pressures of liquid hydrogen, was practically the same as that described in the previous abstract; full details are given in the paper. The pressure on the gas in the thermometer was

directly observed by means of a manometer attached to the apparatus, and was independent of the atmospheric pressure. The temperature of the dead space and mercury column were determined by means of mercury thermometers, and the mean temperature of the vertical portion of the stem immediately above the bulb was measured by means of an auxiliary gas thermometer, with a cylindrical bulb of the same length as that portion of the stem of which the temperature is uncertain.

The coefficient of expansion of the glass of which the thermometer bulbs were made was determined by measuring the contraction of the inner tube of a vacuum vessel, 1000 m.m. long, when filled with liquid air. The coefficient between 0° and 100° had been found to be 0.0000284; between 0° and -190° it was 0.0000218.

The pure hydrogen and helium employed in the thermometric measurements was prepared by methods which are described in the next paper, Part III., Appendices II. and III.

The results of our measurements show that with constant volume thermometers, in which the pressure at the melting point of ice is about 1000 m.m., the temperatures between 80° and 90° Abs. on the helium scale are 0.1° higher than on the hydrogen scale. Olazewski obtained identical readings on the two thermometers. This may be accounted for by the fact that he employed helium from clèveite which had only been purified by sparking with oxygen, and which possibly contained a trace of argon or other impurity. Our helium was probably quite pure, as it had been passed through a coil cooled to 15° Abs. in liquid hydrogen, and was therefore a more perfect thermometric substance than that employed by Olazewski.

This result is further discussed in the full paper.

The Vapour Pressures of Liquid Oxygen.

Pressures in millimetres.	Temperatures on the hydrogen scale.	Temperatures on the helium scale.
800	90.60°	90.70°
760	90.10	90.20
700	89.33	89.43
600	87.91	88.01
500	86.29	86.39
400	84.39	84.49
300	82.09	82.19
200	79.07	79.17

Part III.—By M. W. TRAVERS and A. JAQUEROD.

VAPOUR PRESSURES OF LIQUID HYDROGEN.

The liquid hydrogen employed in these investigations was obtained by a method devised by one of us two years ago, and described in the *Philosophical Magazine*, 1901, vol. xvii., p. 412. About 400 c.c. of liquid hydrogen was employed in each of the seven sets of experiments, of which the following are the results. After filling the gasometer with hydrogen, and collecting the quantity of liquid air (8 litres) necessary to cool the apparatus, this quantity of liquid hydrogen can be obtained in half-an-hour from the moment at which the operations are commenced. Our experience has shown us that when liquid hydrogen is once obtained, it is much more convenient to manipulate than liquid air. As its latent heat of vaporisation is very high, little loss is entailed through cooling apparatus, previously cooled in liquid air, to its boiling-point. Further, the liquid shows no tendency to become superheated, and boils steadily, even under reduced pressure.

Dewar (*Roy. Soc. Proc.*, Feb., 1901, vol. lxviii., p. 40) has obtained the following values for the boiling-point of hydrogen on the constant volume hydrogen scale:— -253.03° , -253.37° , -252.81° , -250.35° ; the pressure on the gas at the ice-point being 287 m.m., 270 m.m., 739 m.m., and 127 m.m. respectively. On the scale of a thermometer filled with helium containing 7 per cent of neon, at a pressure corresponding to 728 m.m. of mercury at the ice-point, he found the temperature to

be 252.68° and 252.84° C. In calculating the temperatures on both thermometers he employed Chappuis's coefficient of expansion for hydrogen, 0.00366254.

In our experiments we have employed the three small thermometers which we used to determine the boiling-point and vapour pressures of liquid oxygen. The thermometers were filled with pure hydrogen or helium, obtained by the methods described in Appendices II. and III. to this paper. The small bulb communicating with a manometer, which had in the former experiments contained pure liquid oxygen for the measurement of the vapour pressure, contained, in these experiments, pure hydrogen from palladium. This method of measuring the vapour pressure was essential to the accuracy of the experiments, for it appeared that the vapour pressure of the pure hydrogen, and of the hydrogen in the vacuum vessel surrounding the thermometer, always differed slightly, probably owing to the presence of impurities dissolved in the latter. The agreement between the results obtained with different thermometers, containing different samples of gas, is indicated in the following table:—

Thermometer.	Vapour pressures of liquid hydrogen. M.m.	Temperature.	
		Found.	From smoothed error.
I. Hydrogen Scale.			
A (12 c.c.)	757.2	20.17°	20.21°
B (26 c.c.)	766.6	20.28	20.25
II. Helium Scale.			
A (12 c.c.)	765.0	20.42	20.44
" "	759.2	20.41	20.41
B (26 c.c.)	770.0	20.43	20.46
C (26.7 c.c.)	749.0	20.36	20.36

The vapour pressures of liquid hydrogen were measured on the hydrogen scale between the boiling-point and a pressure of 100 m.m. of mercury, and on the helium scale between the boiling-point and a pressure of 50 m.m., by a method which is described in detail in the full paper. The temperatures on the helium and hydrogen scales were found to differ to a greater extent than at the temperature of liquid oxygen. The difference, as the following table shows, is from 0.19° to 0.21° over the range of temperature investigated. Considering that the critical-point of hydrogen lies about 35° Abs., while that of helium is probably in the neighbourhood of 10° Abs., this difference is not surprising.

The Vapour Pressures of Liquid Hydrogen.

Pressure in millimetres.	Temperatures on the hydrogen scale.	Temperatures on the helium scale.
800	20.41°	20.60°
760	20.22	20.41
700	19.93	20.12
600	19.41	19.61
500	18.82	19.03
400	18.15	18.35
300	17.36	17.57
200	16.37	16.58
100	14.93	15.13
50	—	14.11

Appendix I.—The melting-point of hydrogen was found to be 14.1° on the helium scale; the temperature given by Dewar in 1901 is 16° (*Roy. Soc. Proc.*, vol. lxviii., p. 360), but an earlier measurement by him gives the melting pressure as 55 m.m. (*Nature*, Sept. 21, 1899). The details of the experiments cannot be entered into in this abstract.

Appendix II.—The pure hydrogen used in our thermometers, &c., was obtained by means of spongy palladium. The method of purifying the gas is given in detail.

Appendix III.—The gas from the Bath wells is not a good source of helium for thermometric purposes, since it contains much neon, and the latter, as we shall presently show, has a considerable vapour pressure at the tempera-

ture of liquid hydrogen, and cannot be completely separated from the helium.

Pure helium is most readily obtained from clèveite gas, which appears to contain only helium, argon, and a trace of krypton. The gas used in our experiments was passed through a glass coil immersed in liquid hydrogen boiling under normal pressure in one case (Thermometer A), and under a pressure of 110 m.m. of mercury in another (Thermometers B and C). This helium was probably very pure.

Appendix IV.—The following values have been obtained for the vapour pressures of solid neon:—

Temperature (helium scale).	Pressure (millimetres).
20°4'	12·8
15°65	2·4

The vapour pressure of the neon did not change after successive portions of it had been allowed to evaporate. This proved that neon is a homogeneous substance.

Appendix V.—From consideration of the periodic relationship between the critical- and boiling-points of the elements of the helium-argon group, it appears probable that the critical-point of helium lies at about 10·5° Abs. and the boiling-point at 6° Abs.; Dewar (*loc. cit.*, p. 364) fixes the former of these points at below 9° or 10°, and the latter at about 5°. In a series of experiments helium was compressed into a tube, one end of which was cooled in liquid or solid hydrogen. At temperatures down to that of solid hydrogen evaporating under a pressure of 5 m.m. of mercury (probably about 13° Abs.), the pressure on the helium was slowly increased to 60 atmospheres. Under all conditions a change of pressure accompanied a change of volume of the gas, and no evidence that liquefaction had taken place could be obtained.

NOTE OF THE MATHEMATICAL EXPRESSION OF THE VALENCY LAW OF THE PERIODIC TABLE, AND THE NECESSITY FOR ASSUMING THAT THE ELEMENTS OF ITS FIRST THREE GROUPS ARE POLYVALENT.*

By GEOFFREY MARTIN, B.Sc. (Lond.).

TAKING as abscissæ the group numbers of the elements as they appear in Mendeleeff's table, and as ordinates the degree of valence, mark out points corresponding to the lowest and highest degree of valence that can be exhibited by each separate element at ordinary temperatures. Join these points, and we get the two straight lines, A, B, C and B, D, intersecting at B at right angles.

Let v represent the valency and n the group number; then the equation to the line A, B, C is—

$$v - n = 0,$$

and the equation to B, D is—

$$v + n - 8 = 0.$$

Combining in one equation these two lines, we get—

$$(v - n)(v + n - 8) = 0,$$

or—

$$v^2 - n^2 - 8(v - n) = 0 \dots (1).$$

This last equation is the mathematical expression of the variation of the valency of any one element with its position in the Periodic Table; at least, for elements of moderate atomic weight and at normal temperatures.

For put n in this equation successively equal to 1, 2, 3, 8, and we get for the valencies—

* By a "polyvalent element," I mean an element that can act with several different degrees of valence towards other radicals.

$n=1$		$v=1$ or 7
$n=2$		$v=2$ or 6
$n=3$		$v=3$ or 5
$n=4$		$v=4$ or 4
$n=5$		$v=3$ or 5
$n=6$		$v=2$ or 6
$n=7$		$v=1$ or 7
$n=8$		$v=0$ or 8

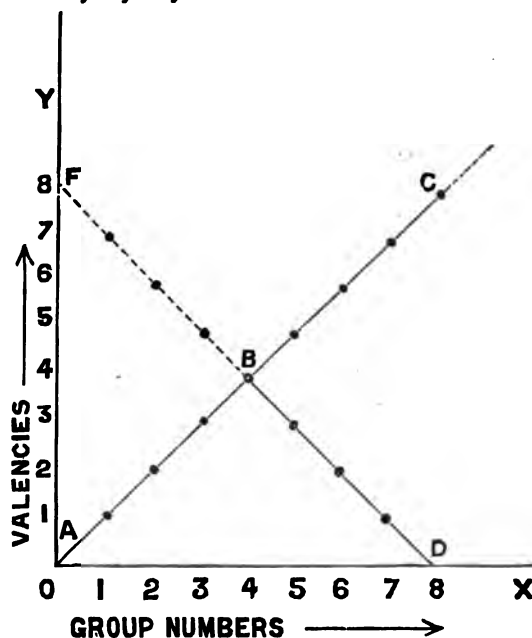
In other words, the degree of valency with which an element can act is—

In Group	VIII.	Lowest.	Highest.
"	VIII.	0	8
"	VII.	1	7
"	VI.	2	6
"	V.	3	5
"	IV.	4	4

So far, we are merely expressing well known experimental facts; but, assuming the law to be true for the first three groups also, we get—

In Group	III.	Lowest.	Highest.
"	III.	3	5
"	II.	2	6
"	I.	1	7

This last part of the law is not at present universally accepted. It implies that the alkali metals, alkaline earth metals, and the elements of the aluminium group can all act with varying degrees of valence, in the same way that the elements of the last few groups can. It is therefore necessary to justify this view.



First, let us consider in what class of compounds would the higher degree of valence be exhibited. A review of the compounds of the elements of the Groups V., VI., VII. shows that they always tend to act with the highest valence when they enter into combination with radicals of a like electrical polarity; but that they tend to act with the lowest degree of valence possible when they enter into combination with radicals or elements which possess an unlike polarity.

For instance, when Cl or I enter into combination with negative radicals, they often act as trivalent elements (e.g., in ICl_3 and the iodonium compounds, $\text{I} \begin{smallmatrix} \text{R} \\ \text{R} \end{smallmatrix}$); while

iodine, there is reason to believe, is even acting with a valence of 5 or 7 in some of the "periodates." Precisely the same phenomena takes place in the case of N, S, and other metalloids, as appears from such compounds as N_2O_5 , SO_3 , SCl_4 , P_2O_5 , &c.

But, in sharp contrast, when union takes place with positive radicals, these same metalloids almost invariably act with their very lowest valence.

For example, the halogens almost invariably act towards metals as monads; likewise oxygen as a dyad in the metallic oxides, and N as a triad in the metallic nitrides.

If, therefore, the same rule holds among the members of the first three groups, we should expect them to exhibit their lowest valence when combined with non-metals (*i.e.*, in salts), and their highest when combined with metals. And this is precisely what we actually find occurring. With such precision, indeed, do the alkali metals and the elements of Groups II. and III. act with their lowest degree of valence towards radicals of an opposite polarity, that even to this very day they are commonly regarded as possessing only one degree of valence, because until recently their only well known compounds were of this nature, notwithstanding the existence of such compounds as KI_3 , $RbBr_3$, $CsBr_3$, $CsBr_5$.

But a new class of compounds has of late been brought to light, in which these metals are united with radicals of the same electrical polarity, *viz.*, other metals.

In these compounds, as was to be expected, they show their polyvalent nature with the utmost clearness, as will be seen from the following formulæ (*vide* Neville, "Report on Chemical Compounds contained in Alloys," *Brit. Assoc. Report*, 1900, 131):—

Na.	K.	Ag.	Zn.	Cd.	Al.
$NaHg_2$	KHg_2	Ag_3Sb	Zn_4Ag	$CdAu$	Al_2Cu
$NaCd_2$	KPt	Ag_2Sn	Zn_2Cu	Cd_2Na	$AlCu$
Na_2Pb		Ag_4Sn	Zn_2Sb	$CdAg_2$	$AlAu_2$
Na_3Bi		Ag_2Al		$CdAg_4$	Al_2Au_5
Na_2Sb					$AlAu_4$
Na_2As					Al_2Cu
Na_4Sn					$AlCu_3$
					$AlAg_2$
					$AlSb$

Whatever valency they possess in these compounds, it certainly is not that which they possess in their compounds with non-metals. Indeed, it appears that here they are acting with a variable valence, and in this are behaving in the precisely same way as the non-metals in their compounds with each other.

That these views are also held by the principal workers in this section of metallurgy is made evident from the following quotations from Neville (*loc. cit.*), the significance and importance of which seems to have escaped the notice of chemists:—

"But as the list now stands it offers matter for the consideration of the student of valency. One sees that the compounds of the metals with the metals present—formulæ that we should expect—from the known valency of the elements, but such bodies as $NaHg_2$, $SnCu_2$, $AlAg_2$ are more remarkable. The first of these is evidently a well marked type, which already occurs several times.

"If I rightly understand Prof. Kurnakov ('Sur les combinaisons mutuelles des métaux'), he thinks that Mendeleeff's law of the total valency of an element for O and H being 8 will find application in the formula of alloys, the H being replaced by other metals. In this case the alkali metals which are monovalent to oxygen should be polyvalent in alloys."

It must be emphasised that this law of valency, as embodied in the equation—

$$v^2 - n^2 - 8(v - n) = 0,$$

only holds in the Periodic Table for elements of moderate atomic weight and at normal temperatures. As the atomic weight or the temperature increases, the deviations from the law become more and more marked. These deviations will be made the subject of a later note.

Consider now some properties of the above equation. If v_1 and v_2 represent its roots, we have—

$$v_1 = n \\ v_2 = -n + 8.$$

Hence,—

$$v_1 + v_2 = 8.$$

i.e., the sum of the highest and lowest degrees of valence with which an element acts towards other elements is a constant whose value is 8.

This is a very remarkable relationship. It includes Mendeleeff's observation that the total valency of an element both towards hydrogen and oxygen is 8. And for this reason:—An element tends to act towards radicals of like electrical sign with its highest valence; but towards radicals of an opposite electrical sign with its lowest valence. Now, hydrogen is electro-positive and oxygen is electro-negative. Therefore by observing the valence exhibited by any one element towards each of these two standard elements, we obtain at the same time the measure of its highest and lowest valence. Hence Mendeleeff's law.

Again, re-arranging the fundamental equation we obtain:—

$$n^2 - 8n + 8v - v^2 = 0.$$

The question naturally arises, does this equation place any restriction on the values v can assume? For from the nature of the case, n , the group number must always be real, and v can only have such values as is compatible with this condition. The condition that n should be real is that—

$$8^2 - 4(8v - v^2) = \text{a positive quantity,}$$

i.e., that—

$$(v - 4)^2 = \text{a positive quantity.}$$

So that no matter what value we give to v , positive or negative, n will always be real.

The answer, therefore, is that the equation imposes no restrictions on the values which v can assume.

Some rather remarkable further developments of this valency law are possible, but are deferred for a later occasion.

University of Berlin, May 31, 1902.

THE ELECTROLYTIC METHOD APPLIED TO URANIUM.*

By LILY GAVIT KOLLOCK and EDGAR F. SMITH.

THE purpose of the present communication is to call attention to the conditions under which uranium can be quantitatively determined in the electrolytic way in solutions of the acetate, the sulphate, and the nitrate (see Tables I., II., and III.), and also to record several separations of uranium by the same means from other metals. It is not necessary to comment further upon the form in which the uranium is precipitated or upon the way in which the deposit is subsequently treated in order to weigh it, as those points have received sufficient attention elsewhere (*Am. Chem. Journ.*, i., 329; *Journ. Am. Chem. Soc.*, xx., 279; and Smith's "Electro-chemical Analysis," p. 94).

It was hoped that possibly iron might be separated from uranium in the acetate solution. Direct experiment demonstrated the opposite. The basic iron salt invariably separated when the temperature of the solution rose to 50° C. Further, the presence of iron in the solution apparently retarded the precipitation of the uranium, as none of the hydroxide of the latter separated with a current of 0.18 ampère and 8 volts. On adding chrome-alum to the uranium acetate solution containing 2 c.c. of free acetic acid and increasing the voltage to 20, there occurred

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xxiii., No. 8.

TABLE I.—*Electrolysis of Uranium Acetate.*

	Uranium present.	29 per cent acetic acid.	Dilution.	Current.	Voltage.	Tempera- ture.	Time.	Uranium found.	Error.
	Grm.	C.c.	C.c.			°C.		Grm.	
1.	0.0986	0.2	125	N.D. ₁₀₇ =0.29 A	16.25	70	5	0.0988	+0.0002
2.	0.0986	0.2	125	N.D. ₁₀₇ =0.3 A	12.2	70	5	0.0989	+0.0003
3.	0.1972	0.2	125	N.D. ₁₀₇ =0.55 A	13.5	70	4	0.1968	-0.0004
4.	0.1972	0.2	125	N.D. ₁₀₇ =0.3 A	10.75	70	6	0.1970	-0.0002
5.	0.1972	0.2	125	N.D. ₁₀₇ =0.135 A	5.5	70	5	0.1966	-0.0006
6.	0.2952	0.2	125	N.D. ₁₀₇ =0.16 A	4.5	75	5	0.2946	-0.0006
7.	0.2952	0.1	125	N.D. ₁₀₇ =0.1 A	4.5	70	7	0.2948	-0.0004
8.	0.2298	0.1	125	N.D. ₁₀₇ =0.09 A	4.25	70	6	0.2297	-0.0001
9.	0.2298	0.2	125	N.D. ₁₀₇ =0.07 A	4.25	70	5½	0.2299	+0.0001
10.	0.2298	0.1	125	N.D. ₁₀₇ =0.05 A	4.0	65	5	0.2299	+0.0001

TABLE II.—*The Electrolysis of Uranyl Nitrate Solutions.*

Uranium present.	Dilution.	Temperature.	Current.	Voltage.	Time.	Uranium found.
Grm.	C.c.	°C.			Hours.	Grm.
0.1222	125	75	N.D. ₁₀₇ =0.035 A	4.6	5½	0.1225
0.1222	125	65	N.D. ₁₀₇ =0.04 A	2.25	7½	0.1218

TABLE III.—*Electrolysis of Uranyl Sulphate.*

Uranium present.	Dilution.	Temperature.	Current.	Voltage.	Time.	Uranium found.	Error.
Grm.	C.c.	°C.			Hours.	Grm.	Grm.
0.1320	125	75	N.D. ₁₀₇ =0.02 A	2	6½	0.1320	—
0.1320	125	75	N.D. ₁₀₇ =0.02 A	2	5½	0.1322	+0.0002
0.1393	125	75	N.D. ₁₀₇ =0.04 A	2.25	5	0.1395	+0.0002
0.1393	125	70	N.D. ₁₀₇ =0.038 A	2.25	7	0.1392	-0.0001

TABLE IV.—*Separation of Uranium from Barium, Calcium, Magnesium, and Zinc.*

U ₂ O ₅ present. Grm.	Barium present. Grm.	29 per cent free acetic acid. C.c.	Dilution. C.c.	Tempera- ture. °C.	Current.	Voltage.	Time. Hours.	U ₂ O ₅ found. Grm.	Error. Grm.	
A.—From Barium (Acetates).										
1.	0.1116	0.11	0.5	125	70	N.D. ₁₀₇ =0.02 A	2	5½	0.1119	+0.0003
2.	0.1116	0.11	0.5	125	65	N.D. ₁₀₇ =0.04 A	8	5½	0.1117	+0.0001
3.	0.1116	0.11	0.2	125	70	N.D. ₁₀₇ =0.1 A	4.5	4	0.1117	+0.0001
B.—From Calcium (Acetates).										
Calcium present.										
1.	0.1116	0.1	0.2	125	70	N.D. ₁₀₇ =0.025 A	2.25	6½	0.1113	-0.0003
2.	0.1116	0.1	0.2	125	70	N.D. ₁₀₇ =0.04 A	2.5	5½	0.1114	-0.0003
3.	0.1116	0.1	0.2	125	70	N.D. ₁₀₇ =0.05 A	2.25	4½	0.1113	-0.0003
4.	0.1116	0.1	0.2	125	70	N.D. ₁₀₇ =0.025 A	2.0	4½	0.1115	-0.0001
C.—From Magnesium (Acetates).										
Magnesium present.										
1.	0.1116	0.1	0.1	125	70	N.D. ₁₀₇ =0.026 A	2.25	6	0.1115	-0.0001
2.	0.1102	0.1	0.1	125	70	N.D. ₁₀₇ =0.05 A	2.25	5½	0.1104	+0.0002
3.	0.1120	0.1	0.1	125	75	N.D. ₁₀₇ =0.15 A	4.0	4	0.1119	-0.0001
D.—From Zinc (Acetates).										
Zinc present.										
1.	0.1120	0.1	0.1	125	70	N.D. ₁₀₇ =0.021 A	2.25	6	0.1120	—
2.	0.1102	0.2	0.2	125	70	N.D. ₁₀₇ =0.017 A	2.25	6	0.1099	-0.0003
3.	0.1102	0.1	0.1	125	70	N.D. ₁₀₇ =0.02 A	2.2	6	0.1100	-0.0002
4.	0.1102	0.1	0.2	125	75	N.D. ₁₀₇ =0.025 A	4.4	4½	0.1103	+0.0001
5.	0.1102	0.15	0.2	125	75	N.D. ₁₀₇ =0.01 A	2.2	6	0.1105	+0.0003
6.	0.1102	0.2	0.2	125	75	N.D. ₁₀₇ =0.02 A	2.25	6	0.1099	-0.0003

no deposition of uranic hydroxide; the chromic oxide, on the other hand, was converted into chromic acid.

Quantitative results were also obtained by the electrolysis of the sulphate. The neutral salt solution was diluted to 125 c.c. and heated to 75° C., when a current of from 0.02 to 0.04 ampère for 107 sq. cm. of cathode surface and 2.25 volts was passed.

The Separation of Uranium from Barium, Calcium, Magnesium, and Zinc.

In the paper by Smith (*loc. cit.*), to which reference has already been made, he calls attention to the separation of uranium in the electrolytic way from the alkali metals and

from barium. Actual results are given. It seemed desirable to amplify the suggestion; hence the presentation of the results given in Table IV. It may be said here that, in attempting to separate nickel and cobalt, no satisfaction could be obtained, so that eventually that particular line of experiment was abandoned. During the precipitation of the urano-uranic hydrate the dish should be well covered, so that as little evaporation as possible occurs. It was observed that in case of evaporation there was danger of other salts separating upon the exposed metal and on re-filling with water the uranium precipitate was apt to enclose the same and thus carry with it a slight impurity. This precaution is especially necessary in the separation from zinc.

VIDAL BLACK AND ANILINE BLACK.

By M. VIDAL.

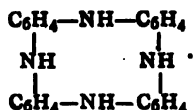
In a previous paper (*Moniteur Scientifique*, September, 1897, p. 655) I described the various phases of the direct formation of my sulphurised colouring materials, and clearly proved the diphenylamine or thiodiphenylamine origin of Vidal black.

I also examined a series of parallel reactions in the formation of aniline black, and I was even able to establish the fact that this latter had, like my black, *p*-amidophenol as a basis.

In fact, if in concentrated sulphuric solution I act on aniline, either by an oxidising agent of a chemical nature, such as bichromate of potassium or by an electro-positive current, I observe in the first place the formation of *p*-amidophenol, which by a graduated hydration of the sulphuric acid, is transformed successively into *p*-amido-oxydiphenylamine and into aniline black; this reaction is also produced with the para-sulphonised derivative of aniline, para-sulphanilic acid, but with the evolution of sulphurous acid.

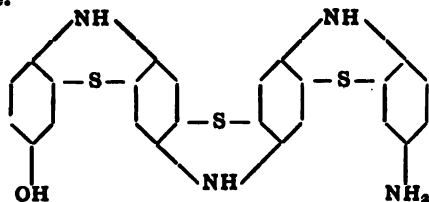
Thus the result of this observation is:—(1) That aniline black has *p*-amidophenol as a basis, this latter being formed in all probability by the transposition of the phenylhydroxylamine obtained in the first place by the oxidation of the aniline in concentrated sulphuric acid; (2) that aniline black, like Vidal black, is the result of the condensation of the diphenylamine radicals.

This almost confirms the structural formula proposed by Goppelsroeder for aniline black:—

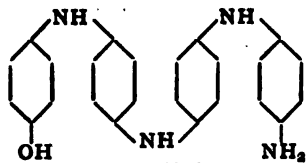


But this formula leaves something wanting; aniline black has the property of becoming green under the influence of certain acid reducing agents, and the green tint thus produced becomes blue in alkaline solution. This fact proves that aniline black should contain one or more active functions, and that the cyclic formula attributed to it by Goppelsroeder will not suffice.

It is necessary to construct a formula with free salifiable groups to explain the property this colouring matter has of being reduced successively under the influence of both reducing and oxidising agents, such as the one I attributed to the sulphurised material para-amido-para-oxydiphenylamine.



Vidal black, from *p*-amido-*p*-oxydiphenylamine.



Aniline black.

Further, the Vidal blacks confirm this hypothesis. While Vidal black from *p*-amido-*p*-oxydiphenylamine has only a slight affinity for alkalis, the Vidal black from *p*-dioxy-diphenylamine, because of its two phenolic functions, combines with soda, giving off a considerable

quantity of heat. But, alike in the case of aniline black as in that of the various sulphur blacks, under the influence of oxidising and reducing agents, and of acids and alkalis, the tints of the colouring materials are either green, blue, or black.

From what has preceded, the analogy of origin, formation, and character appears to be well established between Vidal black and aniline black, and M. Goppelsroeder's formula, with the above mentioned modification, can be applied to aniline black.

Further, the facts I described in my French patent, No. 307,631, January 30, 1901, that is to say, the condensation of *p*-amido-*p*-oxydiphenylamine in the presence of oxidising agents to furnish aniline black has just found an application. According to their patent, No. 313,035, July 27, 1901, the "Compagnie Parisienne des couleurs d'aniline" substitutes *p*-amido-*p*-oxydiphenylamine for aniline to obtain blacks by oxidation.

On the other hand, the piazothiolic formula attributed by M. Geigy to Vidal black and other sulphurised colouring materials is not in accordance with the preceding observations and the methods of production of the different sulphurised materials of the Vidal black series.

The following questions may be asked:—1. How can we explain the formation of the sulphurised derivatives of the thiodiphenylamines with a piazothiolic formula? 2. How can *p*-amidophenol and phenylenediamine give rise to a piazothiolic group, by means of the *p*-dioxy, *p*-diamido, and *p*-amidoxydiphenylamines which they form, when the amido group in the ortho-position with regard to the diphenylamine bond is completely at fault?

Consequently, if the result with aqueous SO_2 at 180° is the same with the diphenylamines having an amido group in the ortho-position, and bodies not able to form diphenylamines possess this essential condition for the piazothiolic formation, it is necessary to reject Geigy's hypothesis for the direct formation of sulphurised colouring materials, especially as the analogies observed between aniline black and Vidal black suffice on their part to destroy it.—*Moniteur Scientifique*, 1902, Series 4, vol. xvi., p. 218.

THE SEPARATION OF GLUCINA.

By G. WYROUBOFF.

THE difficulties met with when we wish to separate glucina from the iron and alumina which always accompany it in emerald, the principal gluciniferous mineral, are well known, especially if we have only to deal with a small quantity of the material.

Carbonate of ammonia dissolves glucina very slowly, and finally dissolves a little alumina if it is not condensed by heat or by prolonged contact with water.

Quite recently (*Bull. Soc. Min.*, 1902, vol. xxv.), while discussing a paper by MM. Rosenheim and Woge on the valency of glucinum, I was led to examine a certain number of its compounds.

Among these compounds one was found which has, however, been known for a very long time, since it was described by M. Debray in his thesis, which is very slightly soluble and which enables us to precipitate almost the whole of the glucina at once. This compound is the oxalate, $\text{Gl}_2\text{O}_3 \cdot 6(\text{C}_2\text{O}_3) \cdot 3\text{K}_2\text{O}$. At 15° 100 parts of water dissolve 1.4 parts of this salt, and the solubility increases only slowly with the temperature. This salt only contains 9.5 per cent of Gl_2O_3 ; it therefore follows that the solubility of the precipitated glucina is about 1 part per thousand.

Now, we know that the oxalates of the same formula containing Al_2O_3 , Fe_2O_3 , and Cr_2O_3 dissolve at the ordinary temperature in two or three times their weight of water.

In this fact there is, as can be well seen, a method of separation, if not quantitative, at least approximate, and, in any case, very rapid. I have endeavoured to apply

this method to emerald from Limoges, which contains, according to an analysis made by M. Lebeau, about 14 per cent of glucina, and I was able to extract a little more than 9 per cent perfectly free from alumina and iron.

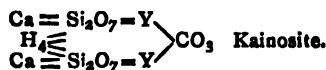
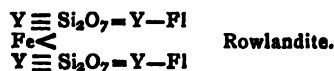
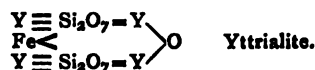
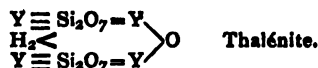
It sufficed to attack the mineral with potash, eliminate the silica in the usual method, evaporate the solution of chlorides to a small volume, and add a concentrated solution of binoxalate of potash. After some time a crystalline precipitate is deposited, which is collected on a filter, and washed with water. The salt, which is very slightly soluble in water, dissolves with ease in dilute acids; it is for this reason that the loss is high, as in the double decomposition, hydrochloric acid is necessarily set at liberty, and remains in the solution.

Perhaps it would be better to neutralise exactly by means of an alkali or to add acetate of soda so as to get better returns, but I have not followed this matter up as it does not really come within the scope of my work, and I only now mention this process of separation because it appears worthy of the attention of those who take an interest in the still so little known compounds of glucina. — *Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 14.

THE COMPOSITION OF YTTRIALITE, WITH A CRITICISM OF THE FORMULA ASSIGNED TO THALENITE.*

By W. F. HILLEBRAND.

In a paper describing the new yttrium silicate thalénite (*Bull. Geol. Inst. Upsala*, 1898, iv., 1), the author, C. Benedicks, casts doubt on the formula assigned by Hidden and Mackintosh (*Am. Journ. Sci.*, 1889, xxxviii., 477) to their mineral yttrialite from Llano Co., Texas, for which they deduced the formula $R_2O_3 \cdot 2SiO_2$ or $R_2Si_2O_7$, in which R_2O_3 includes the sesquioxide equivalents of very considerable percentages of monoxides and dioxides. Benedicks would assign yttrialite to a group of which rowlandite is the prototype and to which he believes thalénite and kainosite belong, being either plain basic salts of $H_2Si_2O_7$ of the type $R''R'''_4Si_4O_{15}$ or derivatives in which the fifteenth oxygen atom is replaced by its equivalent in fluorine (rowlandite) or CO_3 (kainosite) as shown below.



Benedicks says: "Dem Yttrialit, von Hidden und Mackintosh beschrieben, sollte die Formel $R_2O_3 \cdot 2SiO_2$ zukommen, worin R hauptsächlich Ytter- und Thorerde ist. Dabei wird aber ca. 4% Eisenoxydul in der Analyse vernachlässigt. Wird dies nebst etwas Kalk und Bleioxyd mitgerechnet, so bekommt man die Formel $Fe''O, 2R_2O_3, 4SiO_2$, analog mit der des Thalénits, welche besser die Zusammensetzung des Yttrialits wiedergibt, obgleich die Übereinstimmung gar nicht gut ist."

It is, however, not true that Hidden and Mackintosh neglected to take account of the ferrous iron, &c., of their analysis. It was regarded in the derivation of their

empirical formula $R_2Si_2O_7$. How wholly unwarranted was the substitution by Benedicks of his formula for yttrialite is shown by the molecular ratio for $RO : R_2O_3 : SiO_2$, which he gives as 1 : 2 : 4 instead of 1 : 3.25 : 7.42, as calculated by me from Mackintosh's figures, wherein for a sound reason I have converted his UO_3 into UO_2 , thus throwing it with the ThO_2 , where it naturally belongs. The agreement is indeed "gar nicht gut."

Benedicks has made the grave mistake of counting Mackintosh's monoxide bases a second time, thus making a basic salt $R''R'''_4Si_4O_{15}$ instead of the normal one $R''R'''_4Si_4O_{14}$, to which Mackintosh's results closely conform.

Discussion of Benedicks' Formula for Thalénite.

Moreover, in the light of the data furnished by Benedicks himself it cannot be admitted that the formula $H_2Y_4Si_4O_{15}$ for thalénite is established.

Water was determined by him according to Penfield's method (*Am. Journ. Sci.*, 1894, xlviii., 31), but without any hint as to the particular modification employed. If, as seems probable, the water expelled from the mineral was caused to re-condense in the cooler part of the ignition tube, the latter being then weighed and again after driving the condensed water out, two serious sources of error have to be considered: (1) The CO_2 present in the mineral, which would count in part as water unless a very careful correction was made, as provided for by Penfield. No mention is made by Benedicks of any such correction. (2) Nitrogen and helium are said to comprise 1.4 per cent. of the mineral by weight. If so, these would introduce an error in the above water determination of contrary sign to that due to CO_2 , and if the proportion of helium were large this error might be of very considerable magnitude.

In an appendix to his paper Benedicks gives an analysis of what he considers to be a very pure form of thalénite. He makes no comparison of this with his earlier analysis, nor does he deduce a molecular ratio, which I find to be 1 : 2.6 : 5.15, or 1 : 3.03 : 6.02 if small amounts of lime, magnesia, and soda are neglected, instead of 1 : 2 : 4 as required by his formula. There being no CO_2 in this purer material, the value for water (if determined as above surmised) may be supposed to be affected only by the error due to nitrogen and helium. It will be seen that the neglect to regard lime, magnesia, and soda in his second analysis affects the ratio very seriously. This neglect may be justified in figuring on his first analysis because of an approximate balancing by CO_2 , but it would be by no means so in the other in spite of the very satisfactory ratio obtained and leading to the empirical formula $R''R'''_6Si_6O_{22}$, which is susceptible of a variety of interpretations. It may represent a basic salt of diorthosilicic acid $R''R'''_5(R''O)(Si_2O_7)_3$ or of metasilicic acid $R''R'''_4(R''O)_4(SiO_3)_6$, or possibly even of other acids.

Finally, if the values for nitrogen and helium are really anywhere near so great as given, an additional argument against the validity of his formula is furnished. For, in the light of Kohlschütter's recent researches (*Ann. der Chem.*, 1901, cccxvii., 158) and my own less conclusive work of a much earlier date (*Bull. U.S. Geol. Surv.*, 1891, No. 78, pp. 76–78), it is in the highest degree probable that nitrogen and helium are not occluded in uraninite and other minerals, but are in chemical combination. Now, if this is so, in a mineral containing as much as 1.4 per cent. of nitrogen by weight this must, quite irrespective of its form of combination, play so important a rôle in the molecule as to utterly invalidate any formula based on calculations from which it is omitted. If the above percentage is made up in large part of helium, its effect, because of its low atomic weight, must be vastly greater than that of nitrogen.

Until light is thrown on the nature of the combinations these two gases form in minerals, no very positive conclu-

* From the *American Journal of Science*, xiii., No. 74.

sions can be reached as to the formulae to be assigned to those minerals which contain them in more than traces.

Chemical Investigation of Yttrialite.

At the earnest request of Mr. W. E. Hidden, the discoverer of yttrialite, I undertook to re-analyse the mineral in order if possible to settle definitely the question of its composition. This seemed especially desirable since a large quantity of very fine material was available.

The appearance and behaviour of the mineral agreed in all respects except one with those of the original description (*Am. Journ. Sci.*, 1889, xxxviii., 477). It is there stated that the strongly ignited mineral is insoluble in acid. This is a mistake, for when powdered the solubility in hydrochloric acid is even then perfect, although not rapid.

Careful examination of thin sections under the microscope showed a condition that augured ill for decisive analytical results despite the apparently fine quality of the large specimens. Distinctly foreign mineral fragments were as good as absent except for insignificant coatings of a white alteration product, presumably a carbonate, but considerable shading was apparent in the slides, indicative of alteration or intimate contamination in the mass of the mineral itself. However, after treatment with hot dilute hydrochloric acid (whereby much yttrialite was dissolved) followed by dilute sodium carbonate, the clear glassy residue appeared to be improved in appearance and the specific gravity of two samples, each composed of small grains uniform in size for each sample, had risen from 4.596 and 4.590 to 4.654 at 23.3° C. and 4.646 at 26° C. respectively. The two sizes were then mixed, giving one sample of about 4.65 sp. gr. at 25° C. That of the unpurified material analysed by Mackintosh was 4.575.

Qualitative tests showed the absence of zirconium, glucinum, and aluminium and the presence of a little more than a trace of fluorine, of a very little carbon dioxide, besides some other gases which were set free by treatment with acids and by fusion with an alkali carbonate.

Silica was separated by two evaporations with hydrochloric acid; lead was then thrown out by hydrogen sulphide.

The earths when finally collected together free from all iron, manganese, uranium, titanium, phosphorus, calcium, and magnesium, were ignited and weighed. No filtrate from a precipitate of earth-oxalates was ever regarded as free from earths till after evaporating, igniting, and re-testing. This is a needed precaution in all similar analyses.

The combined earths were dissolved in nitric acid and evaporated to dryness. A saturated solution of potassium sulphate was poured upon the dry mass, which wholly dissolved, but almost at once began to deposit double sulphates. Solid potassium sulphate was then added in crystals. After twenty-four hours the precipitate was collected on a filter and washed with the precipitant solution. Twice was this precipitation repeated after first dissolving the double salts in acid, precipitating by potassium hydroxide, washing, re-dissolving in nitric acid, and evaporating. Then only was the extraction of the yttrium-erbium group practically complete. From the combined filtrates the soluble earths were thrown out, re-converted into nitrates, and again treated with potassium sulphate, whereby a little further insoluble matter was obtained. Yttrium-erbium oxides obtained from the oxalates gave a light-coloured mixture of 265.6 mol. wt. (108.8 at. wt.), which furnished a pink nitrate solution showing the erbium absorption bands strongly marked, with very faint indications of the strongest band of the didymium components.

The insoluble sulphates were converted into chlorides and thrice treated with potassium hydroxide and chlorine to precipitate thorium and cerium. From the filtrates the soluble earths were recovered and subjected to a repetition of this treatment. Their oxides after purification were

light dirty brown when heated over the Bunsen flame, but greyish white when blasted. Their mol. wt. was 335.5 (at. wt. 143.7); their nitrate solution was pink and gave the characteristic didymium component absorption bands altogether free from those of the erbium constituents.

Cerium and thorium were separated by a combination of the ammonium oxalate and thiosulphate methods. The weighed thoria was pure white, the ceria of a pale salmon colour.

The condition of the uranium was shown by its precipitability as tetrafluoride on dissolving the mineral in hydrofluoric acid. The reaction afforded a ready means of estimating with exactness the ferrous iron by permanganate after rapidly filtering off the fluorides precipitated in an atmosphere of carbon dioxide. Solution of the mineral in a sealed tube with dilute sulphuric acid allowed of finding the oxygen value of both UO_2 and FeO by permanganate. The result thus found for UO_2 agreed marvellously well with that calculated from the U_3O_8 found gravimetrically in a separate portion.

Analyses of Yttrialite.

	Hillebrand.		Mackintosh.	
		Mol. ratios.		Mol. ratios.
SiO_2	29.63	0.4938	29.17	0.4861
TiO_2	0.05		—	
ThO_2	10.85		12.00	
UO_2	1.64 (a)	0.0470	0.79 (d)	0.0483
Ce_2O_3	3.07		1.86	
La_2O_3 , &c. ..	5.18 (b)		2.94 (e)	
Y_2O_3 , &c. ..	43.45 (c)		22.67 (f)	
Do.		0.1930	5.30 (g)	0.1864
Do.			4.50 (h)	
Do.			14.03 (i)	
Al_2O_3	—		0.55	
Fe_2O_3	0.76		—	
FeO	1.96		2.89	
MnO	0.88		0.77	0.0655
PbO	0.80		0.85	
CaO	0.67	0.0608	0.60	
MgO	0.16			
$H_2O+105\text{ C.}$..	0.32			
$H_2O-105\text{ C.}$..	0.04		0.79	
CO_2	0.11			
P_2O_5	0.12			
N, He } Diff. ..	0.31			
Fl, Alk }				
	100.00		99.75	

- (a) Volumetrically; gravimetrically, 1.62 per cent.
- (b) Atomic weight, 143.8; molecular weight, 335.6.
- (c) Atomic weight, 108.8; molecular weight, 265.6.
- (d) Given as 0.83 UO_2 by Mackintosh.
- (e) Atomic weight, 162; molecular weight, 372.
- (f) Atomic weight, 110.3; molecular weight, 268.6.
- (g) Atomic weight, 110.53; molecular weight, 269.06.
- (h) Atomic weight, 114.9; molecular weight, 277.8.
- (i) Atomic weight, 120; molecular weight, 288.

Nitrogen (?) and helium (?) were obtained quantitatively by fusing the mineral with sodium-potassium carbonate in a current of carbon dioxide, and collecting the gases in a nitrometer over potassium hydroxide. The volume was between 1 and 2 c.c. per gm. of yttrialite. The gases were not further examined.

The analysis is given above, together with that of Mackintosh for comparison.

The ratios of Mackintosh's analysis as calculated above by me are certainly wrong in so far as they are affected by the value for iron, which he assumed to be wholly ferrous. If corrected in accordance with the statement of my analysis, or, what amounts to the same thing for the purpose of illustration and is simpler, if my ratio is altered to conform to his statement for RO and R_2O_3 bases, it becomes 0.0644 : 0.1882, a very close agreement.

It is altogether probable that Mackintosh's separations of the earths were not so far-reaching as mine, and this belief is borne out by the differences in the experimental molecular weights for the lanthanum and yttrium groups, mine being more in accordance with what might be expected, and, moreover, agreeing almost exactly with those which were found by me for rowlandite in 1893, namely, 336.8 and 266.2 respectively.

(The three minerals gadolinite, yttrialite, and rowlandite occur in Llano County in most intimate association, suggestive of close community of origin, a suggestion which is emphasised by the marvellous agreement for gadolinite and yttrialite, not only in the relative proportions of the trivalent earth metals but in their absolute amounts as well.)

		Ce_2O_3	La_2O_3	&c.	Y_2O_3	&c.
Gadolinite (Genth)		2.65	5.22	44.35		
"	"	2.66	5.01	44.45		
" (Bakins)		2.62	5.22	41.55		
Yttrialite (Hillebrand)		3.07	5.18	43.45		
Rowlandite		5.06	9.34	47.70		

This concordant testimony of three analysts may be regarded as strong evidence of the correctness of the earth separations made by them in these cases. Nearly the same relation is shown by the trivalent earth-metals of rowlandite, as seen in the table above.)

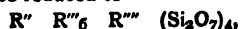
It is, of course, impossible to say what disposition should be made of the small amounts of firmly held water, phosphorus, carbon dioxide, fluorine, and alkalis. The ratios of my analysis are therefore to a slight extent incorrect, but probably not enough to influence any conclusions that may be drawn. One thing is apparent, that the preliminary purification by acid has had no pronounced effect on the composition of the mass acted on, otherwise Mackintosh's and my analyses should show far greater differences in the main constituents.

The crude empirical formulæ deducible from the ratios of the two analyses are nearly—

Hillebrand		R''_{61}	R'''_{386}	R''''_{47}	$(\text{Si}_2\text{O}_7)_{247}$
Mackintosh		R''_{65}	R'''_{373}	R''''_{48}	$(\text{Si}_2\text{O}_7)_{243}$

there being a slight deficiency of oxygen atoms in each case for the radical Si_2O_7 , which is increased by allowing for the CO_2 and P_2O_5 .

In so far then as the character of the acid radical is concerned, the results of Mackintosh's analysis are fully confirmed, and there is absolutely no ground for accepting Benedicks' basic formula, which, as I have already shown, is based on a palpable error. But the ratios are not at all such as to lend themselves to ready re-resolution into isomorphous salts of the acid $\text{H}_2\text{Si}_2\text{O}_7$. By doing quite unwarranted violence to the analytical data the above formulæ might be reduced to—



which can be readily represented structurally as a single complex molecule or as a mixture of molecules like $3\text{R}''_2\text{Si}_2\text{O}_7 + \text{R}''\text{R}''''\text{Si}_2\text{O}_7$. In neither case, however, is the type of the rowlandite molecule approached, which requires an altogether different ratio of monoxide, dioxide, and trioxide bases, nor, if the second be accepted, is it at all clear that the two molecules would be mineralogically equivalent, that is, isomorphous.

An alternative hypothesis is to regard the mineral as a mixture containing the anhydrous thorite molecule. Proceeding on this assumption and deducing all thorium and uranium and the proper amounts of silicon and oxygen, the crude empirical formulæ become—

Mackintosh		R''_{65}	R'''_{373}	$\text{Si}_{438}\text{O}_{1501}$
Hillebrand		R''_{61}	R'''_{386}	$\text{Si}_{447}\text{O}_{1533}$

which may be interpreted as basic salts of metasilicic acid:—

R''_{65}	R'''_{185}	$(\text{R}''''\text{O})_{187}$	$(\text{SiO}_2)_{438}$
R''_{61}	R'''_{194}	$(\text{R}''''\text{O})_{192}$	$(\text{SiO}_2)_{447}$
R''	R'''_3	$(\text{R}''''\text{O})_3$	$(\text{SiO}_2)_7$

or—

this last being easily susceptible of symmetrical representation in graphic form.

On the whole I prefer to leave the constitution of yttrialite unsettled until further evidence can be gathered, either from analyses of allied minerals or from yttrialite itself of more certain purity than any that has yet been discovered.

It must not be forgotten that the gases other than CO_2 contained in the mineral may be the cause of the inability to arrive at satisfactory conclusions in the case of this and all other minerals which contain them, as I have already pointed out.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

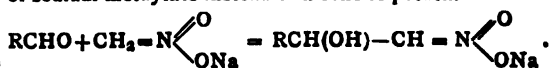
Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxv., No. 1, July 7, 1902.

Electrolysis of Silver Nitrate.—A. Leduc.—A bath of silver nitrate, primarily neutral for example, may generally be said to become more and more acid as electrolysis is pursued, if the anode is soluble. MM. Rodger and Watson find, on the contrary, that the acidity of the bath seems to diminish with use. This contradiction is only apparent, the results depending on certain conditions. The author examines solutions with (1) a platinum anode, and (2) a soluble anode. His experiments show that the contra-electromotive force in a voltameter containing nitrate of silver, a substance which is generally supposed to have a very slight or even no electromotive force, is not in reality negligible. His experiments show that this E.M.F. is about 0.3 volt.

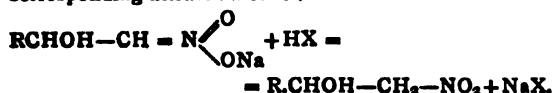
Hydration of Zinc Oxide.—M. de Forcrand.—Anhydrous zinc oxide prepared at 125° is apparently in the least condensed form, and is transformed into normal crystalline hydrate, $\text{Zn}(\text{OH})_2$, evolving $+2.19$ cals. when uniting with water in the liquid state. This is the only result which the author considers to have a precise signification, and it differs from Thomsen's and Massol's results. The condensed anhydrous oxide, prepared at a bright red heat, gives a certain number of different condensation hydrates. The mean value corresponding to the fixation of 1 mol. of water lies between 4.5 and 5 cals. The most hydrated amongst these latter hydrates lose water progressively when they are heated, etherifying themselves and giving meta-zincic acids more and more stable and more and more polymerised. The condensation of $n\text{Zn}(\text{OH})_2$ and its transformation into $(\text{ZnO}, \text{H}_2\text{O})_n$ evolves about $n \times 3.80$ cals. Lime and probably many other metallic oxides give rise to analogous phenomena.

Oxidising Properties of a Pyranol.—R. Fosse.—The author endeavours to prepare hypoiodite of dinaphthopyranoxonium. By analogy with one of the known preparations for the hypochlorite and bromite, he allows hydriodic acid to act on dinaphthopyranol, and was surprised to obtain not the expected hypoiodate but oxonium triiodide. A study of this reaction leads to the conclusion that dinaphthopyranol behaves as an oxidising agent towards HI, and a still more curious example of its oxidising properties is its action on diphenopyranol.

Condensation of Nitromethane with the Aromatic Aldehydes.—L. Bouveault and A. Wahl.—The authors repeat Thiele's experiments on the preparation of nitro-styrolene by performing the condensation by means of sodium methylate instead of alcoholic potash.



These salts are very soluble in water, which does not dissociate them, but acids decompose them, setting free the corresponding nitrated alcohol.



At the moment of its formation, and in varying proportion according to particular cases, this nitrated alcohol is dehydrated, giving the required nitrostyrene.



The authors have also applied this method to condensations of nitromethane with anisic, piperonylic, and ortho-nitrobenzoic aldehydes and with furfural.

Action of Diazoic Salts on Desmotroposantonine and Desmotroposantonous Acid.—E. Wedekind and Oscar Schmidt.—Some years ago M. Wedekind showed that santonic acid combines with diazoic salts in alkaline solution, giving rise to substances having a yellowish red colour, whose constitution was not clearly defined, owing to the instability of these substances and the difficulty of purifying them. On account of these difficulties, the authors now propose to examine the action of the diazoic salts on another isomer of santonine—desmotroposantonine, which was discovered by Andreocci. They succeed in causing desmotroposantonine to combine with the diazoic salts, and obtain a well crystalline and very stable body. Santonine, on the contrary, which does not contain a benzenic radicle, does not combine with the diazoic compounds. In the same way, the authors obtain the azoic compounds of *p*-toluidine, orthonitraniline, *p*-nitraniline, *p*-aminobenzoic acid, sulphanilic acid, and toluidine. All these bodies are well crystallised, have a yellow or red colour, and melt above 260°.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxvii., No. 3.

Form in which Silicon occurs in Cast-iron and in Low Percentage Ferrosilicons.—P. Lebeau.—Already noticed.

Action of some Reagents on Amorphous Silicon.—P. Lebeau.—Already inserted in full.

The Cementation of Iron by means of Silicon.—P. Lebeau.—Already inserted in full.

Alloys of Aluminium and Magnesium.—O. Boudouard.—Already noticed.

Imidodithiocarbonic Ethers. Formation, Constitution, and General Reactions.—M. Delépine.—Iodide of methyl reacts on dimethylformocarbathaldine with the production of diethyllic formal, of iodhydrate of methylamine, and finally of a crystallised iodhydrate of a sulphurised and nitrated base, $\text{C}_4\text{H}_9\text{NS}_2$, which has been examined from the point of view of its decomposition by hydrochloric acid and ammonia; the formula $\text{CH}_3\text{N}:\text{C}(\text{SCH}_3)_2$ was attributed to the base which made it a dimethyllic methylimidodithiocarbonate; that is to say, the most simple trisubstituted term of a new series of compounds having the general formula:—



This constitution was only put forward tentatively, and the author has made a complete research on the question, and now describes numerous and new experiments on these compounds, of which he has prepared ten terms from C_4 to C_{10} .

Imidodithiocarbonic Ethers. Preparation and Properties.—M. Delépine.—To prepare these ethers two molecules of the primary amine are dissolved in two or three times their weight of commercial absolute alcohol in a matras; one molecule of sulphide of carbon is gradually

added; the reaction takes place immediately with the evolution of heat; this is kept down by running water over the matras; as a rule, thiosulphocarbamate crystallises out abundantly on cooling. Two molecules of the halogenised derivative, $(\text{CH}_3\text{I}.\text{C}_2\text{H}_5\text{I})$, are then added, which causes a fresh exothermic reaction; after an hour's time the reaction is over and the liquor barely coloured. On adding 4 or 5 volumes of water nothing is precipitated with the first terms, but, starting from allyl- or propylamine, bad smelling oils are precipitated; these are eliminated by means of ether. The imidodithiocarbonic ethers eventually obtained are mobile, refrangible liquids, perfectly colourless up to C_7 , and slightly yellow above this; they have a powerful odour difficult to define, but something like cooked cabbage for the lower terms. A complete table of the formulae for the ten terms is given, followed by their analyses and compositions.

The β Diketones.—G. Leser.—The author has examined the most diverse β -ketones, and from the properties of all the products obtained he draws some interesting conclusions from the tautomeric point of view; that is to say, the conditions of molecular equilibrium in which these bodies react, either as true di-ketones or as mono- or di-enolic compounds.

Migration of the Methyl Group in the Molecule of Camphor.—E. Blaise and G. Blanc.—Not suitable for abstraction.

On Monoxylbenzalbromindanone.—K. Miniat.—To a warm alcoholic solution of salicylic aldehyde and bromised indanone in molecular proportions, a quantity of caustic soda equal to double the amount of aldehyde used, is added all at once. The liquid takes a red colour for some moments, and then the sodic salt of 2'-oxybenzal-2-bromindanone separates out; this is submitted to repeated crystallisations in alcohol, and brilliant yellow needles are obtained. This body is soluble in warm dilute soda, giving a reddish-yellow colour; on cooling it deposits a pale red crystalline salt. The 2'-methoxy-2-bromindanone is prepared by boiling with acetic anhydride and dried acetate of sodium. The meta-oxybenzal-2-bromindanone is easily formed by following the instructions given for the preparation of the ortho-compound.

Essence of Ylang-Ylang.—G. Darzens.—Wishing to determine the nature of the phenols contained in this essence, and also to discover whether, besides the alcohols already known, other alcohols were present, especially the lower ones of the fatty series, the author took 250 grms. of the pure essence, and instead of saponifying it by means of an alcoholic solution of potash, he effected this by shaking it up in a sealed Wurtz matras with an aqueous solution of potash, the whole being kept hot in a water-bath. After a few moments the solution took a yellow colour, but the mixture was kept at 100° for about ten hours. The aqueous solution was then diluted, and the oily layer washed with water, the washings being added to the aqueous portion. To detect the lower alcohols, the aqueous portion was distilled for a few minutes only, so as to obtain about 10 c.c. only of distillate. This distillate was then saturated with potassium carbonate, when a few drops of a liquid appeared, having the odour and physical appearance of methyl alcohol. These few drops, treated with chloride of benzoyl and carbonate of soda in excess, gave the characteristic odour of benzoate of methyl. The alkaline-aqueous solution was then treated with a current of carbonic acid to remove the phenols, and the cloudy liquid formed was exhausted with ether, and the ethereal solution evaporated in a crucible. The residue was taken up in the smallest possible quantity of soda-lye at 10 per cent, and after filtration shaken up with chloride of benzoyl to transform the phenols into benzoysilised derivatives. The liquid soon solidifies, when an excess of 10 per cent carbonate of soda is added. The insoluble benzoysilised derivative remaining is drained and purified by crystallisation; it was identified as benzoyl-para-cresol. Acetyl-para-cresol was also found.

MISCELLANEOUS.

Appointment.—At a meeting of the Council of the Pharmaceutical Society of Great Britain, held at 17, Bloomsbury Square, on Wednesday August 6th, Dr. William Palmer Wynne, F.R.S., Assistant Professor of Chemistry in the Royal College of Science, South Kensington, was appointed to the Chair of Chemistry in the School of Pharmacy of the Pharmaceutical Society of Great Britain, in succession to Dr. J. Norman Collie, F.R.S., who was recently appointed to the Chair of Organic Chemistry in University College, London.

Institute of Chemistry Examinations.—**Pass Lists** (July, 1902).—**Intermediate Examination**—24 candidates presented themselves; the following (12) passed: W. L. St. J. Alton, Univ. Coll., London, and under M. W. Blyth, F.I.C.; M. Barrowcliff, Univ. Coll., Nottingham; P. H. Carpenter, Finsbury Tech. Coll.; R. W. Clarke, Finsbury Tech. Coll.; O. C. M. Davis, Univ. Coll., Bristol; J. F. P. Fielding, King's Coll., London; G. W. Glen, Glasgow and W. of Scot. Tech. Coll.; B. F. Howard, Finsbury Tech. Coll.; F. T. Jewson; Univ. Coll., Nottingham; W. H. Keys, University of Birmingham; T. H. Lloyd, Univ. Coll., Cardiff; and E. Smith, Glasgow and W. of Scot. Tech. Coll., and under Dr. A. Thomson, M.A., F.I.C. **Final A.I.C. Examination—Branch "A" (Mineral Chemistry)**—7 candidates presented themselves; the following (3) passed: A. E. Garland, Assoc. Royal Coll. Science, London; J. L. Garle, Univ. Coll., London; G. C. Jones (for Fellowship, F.I.C.), Assoc. City and Guilds Institute, London. **Branch "B" (Metallurgical Chemistry)**—2 candidates presented themselves and passed: W. B. Parker, University of Birmingham; J. C. Sheldon, University of Birmingham, and under A. E. Tucker, F.I.C. **Branch "C" (Physical Chemistry)**—2 candidates presented themselves; 1 passed: H. Bassett, B.Sc. (Lond.), Univ. Coll., London. **Branch "D" (Organic Chemistry)**—6 candidates presented themselves; 1 passed: S. Andrews, B.Sc. (Lond.), Assoc. Royal Coll. Science, London. **Branch "E" (Analysis of Food and Drugs, including Examination in Therapeutics, Pharmacology, and Microscopy)**—8 candidates presented themselves; the following (5) passed: G. J. Alderton, King's Coll., London; R. Clegg, B.Sc. (Aberdeen); E. V. Jones, University of Birmingham; W. H. Roberts, M.Sc. (Vit & J), University Coll., Liverpool; H. P. Stevens, B.A. (Oxon.), Ph.D. (Heidelberg), (for Fellowship, F.I.C.). The Examiners in General Theoretical and Practical Chemistry were Dr. Bernard Dyer, F.I.C., and Dr. W. Palmer Wynne, F.R.S., F.I.C. The Examiner in Therapeutics, Pharmacology, and Microscopy was Dr. Arthur P. Luff, F.R.C.P., F.I.C.

The Rhodium Alums and the Separation of Rhodium from Iridium.—A. Piccini and L. Marino.—The authors have succeeded in obtaining rhodium alums, $\text{SO}_4\text{M}_2(\text{SO}_4)_2\text{Rh}_2 + 24\text{H}_2\text{O}$, in regular, yellow octahedra, sometimes having facets of rhomboidal dodecahedra (and even in cubes). They dissolved the hydrate of the sesquioxide of rhodium in dilute sulphuric acid; this gave a bright yellow solution, to which they added an alkaline sulphate, but not in excess or double sulphates would be formed; the solution containing a certain proportion of free sulphuric acid was evaporated in the desiccator, and soon deposited crystals. The caesium salt is one of the most easy to obtain, as it is the least soluble, though much more so than the aluminocæsic alum; it fuses at $110-111^\circ$, giving an orange-coloured liquid; when heated more strongly it loses a portion of its water, and becomes brownish. The rhodium alum closely resembles it, but is more soluble; the potassium salt is still more soluble, and the crystals are very difficult to obtain pure. The ammonium alum, although very soluble, is easily obtained in the form of orange-coloured octahedric crystals, of which the facets attain the size of 1 c.c. square; it is fusible at $102-103^\circ$; on calcination it leaves rhodium

mixed with a little sulphate. Thallium alum is also obtained. Rhodium is the only one of all the metals of the platinum group which forms alums; if the rhodium contains iridium, the crystals of the alum are quite free from this metal. This provides a new method for the separation of rhodium and iridium.—*Zeit. Anorg. Ch.*, vol. xxvii., p. 62.

NOTES AND QUERIES.

*. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Chemical Refuse.—I should be glad if any reader could give me a reply as to whether brewery waste waters, &c., could be considered as chemical refuse.—**ENQUIRER.**

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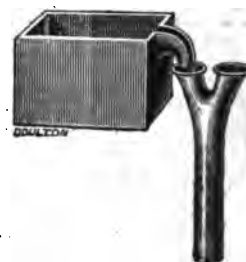
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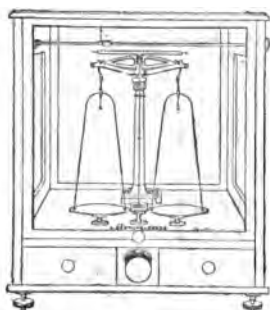
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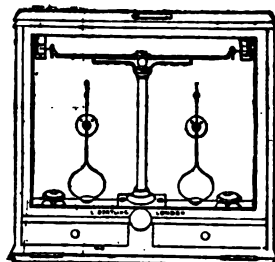


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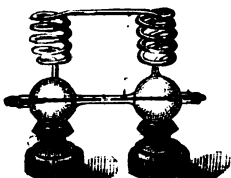
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ON TERBIUM.

By R. MARC.

Of all the rare earths which have yet been described, the existence of the terbium earth has been most disputed. While French authors, as Marignac (*Ann. Chim. Phys.*, xiv., 247), Lecoq de Boisbaudran (*Comptes Rendus*, cii., 395, 483), Delafontaine (*Ann. d. Chem.*, cxxxiv., 99; cxxxv., 188), and others declare in favour of it, others, such as Bahr and Bunsen (*Ann. d. Chem.*, cxxxvii., 11), Krüss and Hofmann (*Zeit. für Anorgan. Chem.*, iv., 27), call it in question. The atomic weights given to terbium by the several authors vary considerably. Thus, Delafontaine gives 142.5; Marignac, 161; Lecoq de Boisbaudran, 159.48; Crookes, 148.5—149.5.

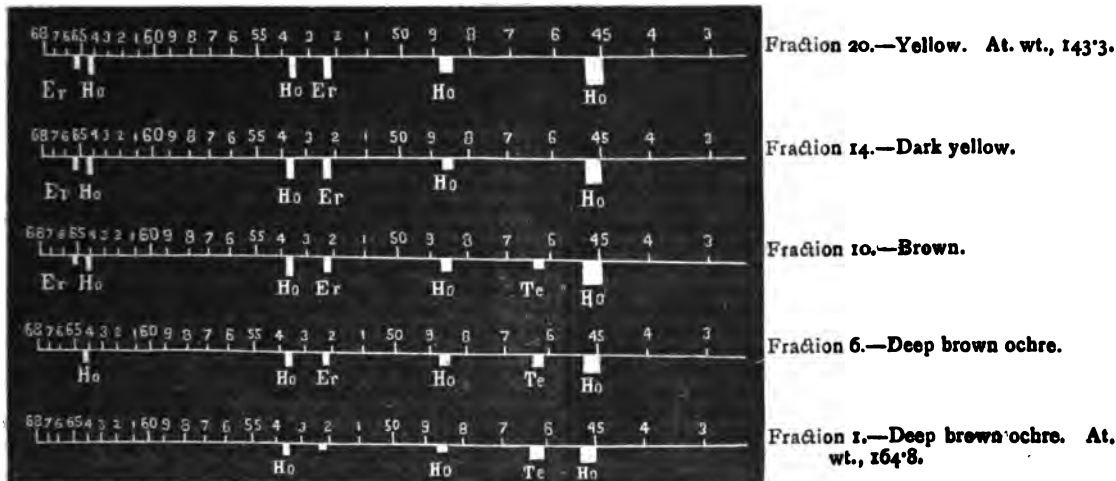
The following characteristic properties are specified:—A yellow colour, which disappears in a current of hydrogen, colourless salts, no absorption spectrum. Only

1. Terbium oxide possesses a deep brown ochre colour. 2. The earths previously known as terbium earths are mixtures of yttria with another substance, which is much heavier, colourless, and has no spectrum (probably ytterbium); these mixtures are coloured by small quantities of terbium oxide.

3. Terbium forms two oxidation products, of which the higher is coloured and the lower colourless (like praseodymium).

4. Terbium probably possesses an absorption spectrum, which consists essentially of the bands λ 464—461.

My material consisted of the lighter coloured oxides, which were separated by Dr. A. Weiss in the preparation of didymium from monasite by the chromic acid method of Muthmann and Böhm (*Berichte*, 1900, xxxiii., 42—49), and the last fractions were certainly the richest. The oxides had a yellow colour, and showed in the absorption spectrum bands of erbium, neodymium, and samarium. After neodymium and samarium had been removed by treatment with potassium sulphate, according to Betten-dorf's method (*Ann. d. Chem.*, cclxx., 376), the residue, which only showed the bands of erbium, was fractionated with ammonia, when the oxides obtained from the alkaline solution became, fraction by fraction, darker, and finally deep brown coloured, while the erbium spectrum disappeared gradually from them, and at the same time a



Delafontaine asserts that the salts are sometimes of a faint amethyst colour, and that the solutions of the salts possess a spectrum, which is composed of the following three bands:—

- λ 522
- λ 497?
- λ 495 (strong)

Krüss and Hofmann proved that the yellow oxide, which Marignac and Lecoq regarded as a compound of terbium, is not a single substance. A yellow earth, prepared according to the directions of these two authors, which had an atomic weight of 158.4 and could not be decomposed by the method of separation used by Marignac and Lecoq, they split up by means of a new method of separation into a series, the first member of which had an atomic weight of 152.4, and the last 163.

We also by a special method of separation have succeeded in decomposing into a series of 4 members an earth, which corresponded to the preparations of terbium of the above authors, and possessed an atomic weight of 158; the atomic weights of the first and last members of the series were respectively 152 and 161. Exhaustive researches have, however, led me to interpret this result differently from Krüss and Hofmann. My results may be shortly stated as follows:—

new band λ 464—461 became visible, and increased in intensity. After a very long series of fractionating I succeeded in almost entirely getting rid of the erbium spectrum, while the band λ 454—449 was still fairly distinct, and the bands λ 640, 536, and 522 were only faintly visible. These four lines, according to Soret, are characteristic of holmium. The band λ 464—461 was clearly visible.

The dark colour of the material was very surprising; as spectroscopic examination showed that it was quite free from didymium, the colour must have been due to another earth. Hitherto terbium has been described only as dark yellow. But as the method of preparation from the erbium-holmium material by means of ammonia would allow of terbium being included, it might be supposed that the latter was present in a higher degree of purity. The atomic weight of this fraction was therefore determined. It amounted to 158 for R_2O_3 .

Thus, this number was similar to that ascribed to terbium by previous authors—Marignac and Lecoq—which seemed singular, considering the great difference in the colour of the materials.

I now endeavoured to determine whether an alteration in the atomic weight would be produced by further fractionating with ammonia. However, this was not the

case. Next I tried to fractionate with oxalic acid. This was done by adding hot concentrated oxalic acid to the concentrated solution of the earths in very strong hydrochloric acid in a little flask, until the solution became slightly cloudy; then the flask was cooled rapidly, so that a very fine crystalline precipitate of oxalate was formed. Thus I succeeded by four such fractionatings in decomposing the earth in such a way that the first member had an atomic weight of 161.18, and the last 151.93. Thus this decomposition corresponds, as has already been pointed out, to that effected by Krüss and Hofmann.

The colour of the oxide in fractions 1 and 4 was approximately unchanged. This circumstance, together with the fact that Marignac's and Lecoq's earths with equal atomic weight were yellow, leads us to infer that the coloured earth cannot have great influence on the atomic weight of the preparation; that is, that it can only be present in small quantity. This supposition was confirmed by the following facts:—

Lecoq (*Comptes Rendus*, cii., 359, 483) had already observed the formation of the superoxide of terbium, and determined the mass of the earth which is converted into superoxide. This quantity, according to his determinations by the method of calcination, amounts to 0.71—0.69 per cent of the total earth.

On dissolving my preparation in hydrochloric acid I observed an evolution of chlorine, and made use of this action to determine by titration the quantity of the earth which is converted into superoxide. By adhering to certain conditions, further specified below, I succeeded thus in obtaining nearly constant results, which were as follows, for the earth with atomic weight 158.

I. 0.9795 grm. oxide; 0.7 c.c. $\frac{1}{16}$ N $\text{Na}_2\text{S}_2\text{O}_3$ = 0.00056 grm. O = 0.058 per cent O.

II. 0.7040 grm. oxide; 0.59 c.c. $\frac{1}{16}$ N $\text{Na}_2\text{S}_2\text{O}_3$ = 0.00047 grm. O = 0.066 per cent O.

Now, if we assume that the formation of superoxide takes place according to the equation $\text{R}_2\text{O}_3 + \text{O} = \text{R}_2\text{O}_4$, we get, taking for R the value 158 obtained by me, the mass of the earth which is converted into superoxide equal to 1.37 per cent and 1.56 per cent, thus on an average about 1.48 per cent.; that is, about twice the quantity which Lecoq found for his darkest earths.

The considerable difference in the two determinations is to be explained by the peculiar behaviour of the earth on calcination, which I shall discuss below.

In a current of hydrogen, the brown earths lost their colour. Now, if the brown colour was caused by the terbium contained in them, a loss of weight must occur on decolorisation in a current of hydrogen, which would correspond to the amount of superoxide determined by titration. After a suitable method, which will be described hereafter, had been discovered, determinations of the material with atomic weight 151.93 were performed by titration and in a current of hydrogen; these agreed fairly well.

The mean of the determinations by titration was 0.073 per cent O, and by reduction 0.069 per cent O, whence the masses of the earth converted into superoxide for the atomic weight 151.93 come to 1.68 per cent and 1.58 per cent respectively. These results confirmed my supposition that the substance to which the colour is due is contained in the material in question in small quantity only—in my case about 14 per cent—and exercises no appreciable influence on the atomic weight.

That such a small quantity should have so great an effect on the colour is not unusual. For instance, if 2 per cent iron oxide is added to yttria, the mixture dissolved, precipitated, and ignited, a deep red-brown mixture is obtained. Similarly 1—2 per cent praseodymium oxide colours chalk under the same conditions an intense brown. One-tenth per cent manganese dioxide gives to chalk a distinct brown colour; one-tenth per cent praseodymium behaves similarly; while in 10 per cent solution a stratum 5 c.m. thick of this mixture yields no spectrum. This is

probably the reason why earlier investigators have not observed the terbium band λ 464—461.

The hydrous salts of my terbium preparations were coloured, thus agreeing with Delafontaine's observations, only the colour must rather be described as flesh colour. Probably the colour of the terbium salts is itself very intense, as is the case with praseodymium salts, which, when present to the extent of about 1—2 per cent, colour the salts of colourless earths with almost the same intensity.

We now return to the behaviour of the terbium preparations, and have to describe the methods which were employed for the titration and determination in a current of hydrogen.

When dry or slightly moistened, the preparations on addition of hydrochloric acid give off chlorine. If, on the other hand, they are suspended in much water, no perceptible evolution of chlorine occurs until they are heated to boiling.

I used the following method for titration:—The dry oxide was moistened with a few drops of water, and some cubic centimetres of a mixture of concentrated hydrochloric acid and potassium iodide solution were added, and the whole allowed to stand in darkness for an hour. The iodine set free was then titrated with thiosulphate.

The behaviour of the earth on igniting presented a difficulty, for the weight first increased a little and then decreased, and only became approximately constant on very strongly heating.

For the determination of the terbium superoxide in a current of hydrogen, the ordinary method, i.e., heating the oxide in a little boat, could not be used, as the reduced oxide attracts moisture with great energy and increases in weight while it is being taken out of the tube and weighed. But by more protracted action of the hydrogen in a Rose's crucible complete reduction may be effected. Therefore I made the determinations in a Rose's crucible, which fitted into a weighing tube with an air-tight stopper. The crucible and weighing tube were weighed together. After reduction the whole was allowed to become quite cool in a current of hydrogen, the hydrogen was expelled by a current of carefully dried air, and the crucible immediately shut up in the weighing tube. To avoid errors, the crucible had to be filled with dry air when weighed before reduction. During reduction the crucible must be strongly heated with a Bunsen burner. On heating moderately decolorisation results with an increase of weight, which peculiarity is to be explained by the fact that the water formed on reduction at once enters into combination, forming a hydrate.

The increase begins simultaneously with change of colour at 230° and increases up to 350°. At this temperature no further increase takes place. At a red heat the decrease begins; at a bright red heat the weight becomes constant. The maximum increase amounts to 0.0089 per cent, the maximum decrease to 0.07 per cent.

For oxidation it is sufficient to ignite in air, but to obtain constant values the ignition had to be performed with a good blowpipe burner. On igniting in a current of oxygen no further increase of weight took place. The determinations had to be made with a fairly considerable quantity of material (not less than 3 grms.), as otherwise it was impossible to detect the increases by weighing.

There has always been much discussion about the position of the rare earths in the Periodic System. This question has been solved with any degree of certainty only in the cases of a few, e.g., scandium, yttrium, lanthanum.

During my work I have noticed that it seems possible that the other earths may be collected, according to atomic weight, into certain groups, as is the case in the ninth group of the Periodic System. The accompanying plan will illustrate this. (See next column).

The behaviour of the oxides on fractionating also confirms the grouping.

If a raw material is decomposed by means of potassium sulphate into two parts, about half the double

I. More Feebly Basic than Yttria.

Ytterbium	Holmium	Erbium	Terbium
173	164 (166-160)		?

II. More Strongly Basic than Yttria.

Gadolinium	Samarium	Neodymium	Praseodymium
156	150	143	140

If we compare the properties of any two elements in a vertical column, we arrive at the following results:—

Ytterbium ..	Oxide colourless	
Gadolinium ..	Colourless salts	No spectrum
Holmium ..	Oxide yellow	Absorption spectrum
Samarium ..	Light yellow salts	
Erbium ..	Oxides rose coloured and dove grey	Absorption spectrum
Neodymium ..	Red salts	
Terbium ..	Oxides deep brown	Absorption spectrum
Praseodymium	Coloured salts	

Form two oxidation products.

salt is precipitated, and the rest remains in solution, so that Series I. is found chiefly in the solution and Series II. chiefly in the crystals. In the case of Series II., the solubilities of the double salts in a saturated solution of potassium sulphate have been determined by Delafontaine (*Comptes Rendus*, xc., 221), and amount to about—

1 : 150-200 for Gadolinium (Ya) potassium sulphate.
1 : 2000 " Samarium " "
Almost insoluble, Didymium " "

(Note.—Bettendorf (*Ann. d. Chem.*, cclxx., 376) also determined the solubility of pure gadolinium in a saturated solution of potassium sulphate by introducing the earths in the form of a concentrated neutral solution of nitrate into saturated potassium sulphate, and allowing to stand for three days. He found 0.76 gm. oxide to 100 parts by volume of solution. I also prepared pure gadolinium (atomic weight, 156.33), and employed the potassium sulphate method, but I did not let the potassium solution stand, but shook it in a shaker for three days. By this method the solubility was found to be decidedly smaller. In 500 c.c. potassium sulphate solution it amounted to 0.44, 0.45, 0.47, 0.47, 0.46. Thus the mean was 0.46, which represents a solubility of less than 1 : 1000. Thus it is clear that the solutions become strongly supersaturated on standing without agitation).

The order of the members of the series becomes apparent on examining the rapidity with which the double salts are precipitated from a saturated solution of potassium sulphate. Praseodymium is precipitated at once; then neodymium; samarium, after from ten to twenty-four hours; and, finally, after shaking for some days, gadolinium.

The potassium double sulphates of Series I., on the other hand, are very easily soluble in potassium sulphate. Their behaviour towards ammonia determines their order. The order of their basicity, as far as can be deduced from the changes of atomic weight and the spectra in the several fractions, is ytterbium, erbium, holmium, terbium.

Arguing from analogy, I should infer from the above table that the atomic weight of terbium is 157.

An article by Biltz has recently appeared (*Berichte*, 1902, xxxv., 562), in which he proposes to arrange the elements of the ninth group in the eighth group, always denoting the three connected elements by the symbol of the best known of them with a preceding X. Thus, XFe would be the symbol for the elements (Mn, Fe) Co, Ni; XPd for Ru, Rh, Pd. In the same way, he recommends collecting the elements of the rare earths as follows:—(La, Ce) Pr, Nd, denoting them by XCe. He expects that in time it will be found possible in this way to arrange in order in the Periodic System the remaining elements of the rare earths. The grouping mentioned by me above

seems to admit of the possibility of such an arrangement. Then the two following groups—

(La, Ce)	Pr, Nd, Sa, Gd
	Te, Er, Ho, Yb

with atomic weights—

(138, 140)	140, 143, 150, 156
(157?)	164, 173
	(160-166)

would be denoted by XCe and XEr, and put in the place of lanthanum and ytterbium. Whether cerium, as Biltz assumes, really belongs, not to Group IV. between zirconium and thorium, but to Group III., may be left undecided.

If now we examine from the above point of view the earth containing terbium which we have described, there can be no doubt that it consists of a mixture of ytterbium earth and yttria, coloured by 1½ per cent of terbium oxide. Unfortunately, there was not sufficient material to isolate the ytterbium earth. On the other hand, I was able by a series of 20 fractions so to split up the earth containing terbium (12 grms. in all) that the first member had an atomic weight of 164.8, and the last 143.3. The spectra of fractions 1, 6, 10, 14, 20 were examined, and are reproduced in the diagram given on page 73.

Fraction 1 had the weakest holmium spectrum, while the lines λ 464-461 were very strongly developed. This fraction was also the darkest coloured. Fraction 19 was comparatively light in colour and showed the holmium spectrum fairly distinctly; next to it two erbium bands, but scarcely any indication of the band λ 464-461. The spectra of the other fractions were intermediate between these two.

Cleve (*Comptes Rendus*, xcl., 328, 381) in his article on erbium earths described a third component of erbium, which he named thulium, and which is said to have an atomic weight of 171. From a great number of different erbium materials I have prepared tables of comparison for spectrum analysis and have examined a long series of fractions, but have not been able to discover anything which would lead us to infer the presence of a third component in erbium. Hence, I think that Cleve's thulium was a mixture of ytterbium and yttrium adulterated by varying quantities of holmium and terbium, just as the decipium oxide belonging to the second series of the above system is probably to be regarded as a mixture of gadolinium oxide and yttria, coloured by praseodymium. Lecoq de Boisbaudran (*Comptes Rendus*, lxxxix., 516) has already proved that philippium, and Soré's X are identical with holmium, and mosandrium with terbium. As regards Demarcay's (*Comptes Rendus*, cxxii., 1484) recent announcement of the discovery of europium, which is characterised by its spectrum of cathode luminescence, that may be explained by the experiments on the spectra of cathode luminescence lately published by us (*Berichte*, 1901, xxxiv., 2460).—*Berichte*, 1902, xxxv., 389.

Sub-oxide of Lead.—S. Tanatar.—The author has taken up the examination of the sub-oxide of lead obtained by Boussingault by the calcination of oxalate of lead. The product formed always contains more lead than it should according to the formula Pb_2O ; this is no doubt due to the reducing action of the atmosphere of carbonic oxide. But if the operation is carried out in a current of carbonic acid, at a well regulated temperature as low as possible, a fine greyish-black powder is obtained, unaltered in dry air, insoluble in, and unchanged by, water, but decomposed by heat, or by alkalis and acids, into metallic lead and PbO . On analysis the material gives 47.72-48.90 per cent of lead (calculated for Pb_2O , 48.14 per cent). The calorimetric examination of the attack with acetic acid, and that of its density, viz., 8.342, shows that we have really to deal with a true sub-oxide of lead, Pb_2O , and not with a mixture of Pb and PbO . If during its preparation, the desired temperature is exceeded, a greyish-green powder is obtained which consists of $Pb + PbO$.—*Zell. Anorg. Ch.*, vol. xxvii., p. 304.

AMMONIUM AMALGAM.

By HENRI MOISSAN.

THE curious experiments of Leestock and Tromadorff, of Berzelius and Pontin, as well as those of Sir Humphry Davy, establish the existence of a so-called ammonium amalgam without definitely showing its composition. This amalgam is obtained by gently shaking up pasty sodium amalgam with a saturated solution of hydrochlorate of ammonium. The reaction has been represented by the following equation:—



The accuracy of this formula has not been verified either by the weights of the materials used or by the volumes of the gases given off.

According to the researches of Gay-Lussac and of Thénard, this amalgam prepared in an aqueous solution of hydrochlorate of ammonium contains $2\frac{1}{2}$ volumes of ammonia for each 2 volumes of hydrogen.

By regarding the ammonium as the radical of the ammoniacal salts, and giving it the symbol NH_4 , it ought to contain a volume of ammonia, double that of the hydrogen, according to the equation:— $(\text{NH}_4)_2 = 2\text{NH}_3 + \text{H}_2$.

The figures given by Gay-Lussac and Thénard are decidedly different to these theoretical figures; however, there is nothing surprising in this, considering the difficulties of these experiments.

After having prepared ammonium amalgam by electrolysis, Landolt observed that on decomposition it gave 2.15 to 2.40 volumes of ammonia gas for 1 volume of hydrogen. But he also noticed that this amalgam did not bring about any double decomposition with the salts of copper and silver, that it differed from the amalgams of potassium and sodium, and that while containing the group NH_4 , it ought not to be looked upon as a metallic amalgam. As a matter of fact, we are not much further advanced to-day than at the time when Landolt published his paper. We are still asking whether the ammonia and the hydrogen are combined in this amalgam, forming NH_4 , or whether these two gases are present in the form of an emulsion in the mercury.

Numerous researches have been made on this subject, and delicate measurements have been carried out without any definite conclusion being arrived at. Further, ammonium amalgam has only been examined, up to the present, at the moment of its decomposition. At the same time, interesting researches have been carried out on new compounds, the ammonium metals of which we have spoken in previous papers.

Other researches were attempted on the solubility of different bodies in liquid ammonia. In this connection we may refer to the work of Seeley, of Gore, and of Franklin and Krauss.

Finally, in some very curious experiments, C. Frenzel, taking up the question at its true starting-point, tried first of all what the conductivity of the ammoniacal liquid was. He showed by means of his delicate experiments that this conductivity diminished with the purity of the ammonia, and that it was comparable to that of water. But to sum up, in spite of these experiments and other researches, among which we may mention those of Le Blanc, Goodwin, Kay-Thomson, Pocklington, and Cohen, ammonium has not yet been isolated.

This question of the reaction of an alkaline amalgam on an aqueous solution of an ammoniacal salt is more complex than it has yet been considered to be.

M. Berthelot has already called the attention of chemists to the part played by alkaline amalgams, and particularly to the action of the definite amalgam, Hg_{12}Na , of M.M. Krant and Pope in solution in mercury.

We know that these amalgams are used in organic chemistry as hydrogenising agents, for fixing hydrogen by inverse substitution on chlorinated bodies, for example, or for transforming the aldehydes or the ketones into alcohols (Friedel and Würtz). M. Berthelot has pointed

out that during these hydrogenising reactions, these amalgams always give off more heat than is given off by the corresponding amount of free hydrogen. This thermic excess joined to the particular action of the reactions developed by the alkalis, accounts for what has up till now been called the nascent state. This interpretation was put forward by Berthelot in 1865.

In taking up the examination of the so-called ammonium amalgam, the first question to be solved consists in accurately measuring the volumes of hydrogen and ammonia given off by the amalgam at the moment of its decomposition. But, on the other hand, the solubility of the ammonia in water is so great that none of the experiments attempted in the presence of this liquid were beyond criticism.

The desiccation of a pasty mass already in the act of decomposing, by means of filter-paper, is absolutely illusory. It is therefore necessary to produce this amalgam in another liquid than water.

This result has been obtained by reacting with chloride of ammonium, in solution in liquid ammonia, on sodium amalgam. The experiment is extremely simple, and is made at -35° ; chloride of sodium is obtained, slightly soluble in the excess of ammonia, as well as a stable metallic mass which increases in volume as soon as it reaches the ordinary temperature.

We first made quite sure that the sodium amalgam kept for several hours in the presence of liquid ammonia between -35° and -39° produced no reaction. We then placed the pasty sodium amalgam in the presence of liquid ammonium into which a small quantity of perfectly dry iodide of ammonium had been dropped. Under these conditions the amalgam reacted on the ammoniacal salt and became more fluid, but without giving off any gas. After prolonged shaking, the amalgam was washed several times with liquid ammonia, and decanted to remove the iodides of ammonium and sodium.

The metallic mass is then cooled down to -80° . At this temperature it appears as a very hard but easily manipulated metal. It is then removed from the vessel in which the reaction has been effected, washed several times with dry ether cooled down to -80° , and saturated with hydrogen. This piece of metal is then placed in a small glass tube, and sealed to a mercury pump. A vacuum is produced, still keeping the temperature down to -80° or -90° . Under these conditions an excellent vacuum can be maintained without any decomposition taking place.

The experiments were made possible by the fact that the so-called ammonium amalgam is able to be formed at -38° or -39° in liquid ammonia without undergoing any decomposition. We made certain that no gas was given off at the moment of the reaction.

So that the double decomposition might be produced with facility, the ammonia was allowed to reach its boiling-point, -33.5° , and if the ammonium amalgam is not too concentrated only a small quantity of ammonia is collected, entirely soluble in water.

When the ammonium amalgam is thus enclosed in a glass tube at a temperature of -80° it is allowed to decompose slowly, gradually reaching the ordinary temperature. At about -40° the mass begins to exude a few drops of mercury; it then takes a liquid form, and as the temperature rises a slight swelling begins to take place. At -30° the mass increases in volume in a very distinct manner, and at $+15^\circ$ it almost entirely fills the glass tube, occupying a volume twenty-five to thirty times greater than the original volume of the amalgam.

During this decomposition it is remarked that the temperature of the tube rises to 5° or 6° above the surrounding temperature. This evolution of heat was constant in all our experiments.

The evolution of gas goes on thus in the vacuum which must be constantly maintained for twelve to fifteen hours. It is very easy to make the decomposition more rapid by heating the amalgam by means of a spirit-lamp.

In some experiments we substituted washing with liquid sulphurous acid or ether saturated with hydrochloric acid gas for the washing with ether.

The results obtained were the same as far as concerns the proportion of ammonia and hydrogen, although under these conditions a certain amount of the amalgam was destroyed. If the gas collected is all put together and analysed, we find that it consists of two volumes of ammonia for one of hydrogen. This is plainly shown by the figures given below:—

Experiment.	Total volume.	H.	NH ₃ .
	C.c.	Per cent.	Per cent.
1	294.72	33.3	66.7
2	264.82	33.4	66.6
3	121.00	33.9	66.1
4	102.31	33.3	66.7
5	528.00	33.3	66.7
6	48.73	33.3	66.7
7	74.82	33.7	66.3

The ammonia was collected in water and titrated or weighed in the form of hydrochlorate of ammonia. The hydrogen was estimated by means of the eudiometer.

In this series of analyses the radical NH₄, corresponding to 2 volumes of ammonia, and 1 volume of hydrogen, would thus seem to exist in the metallic mass prepared at -39°; we are inclined to think, however, that such is not the case. In reality, the hydrogen of the hydriodic acid would appear to furnish a simple or double ammoniacal metallic hydride, and it is the decomposition of this hydride that produces the increase in bulk.

This new interpretation which we are still studying can be applied to the following experiment:—If we react with pasty sodium amalgam on ammonia, we get a slow disengagement of hydrogen without any swelling up. On the other hand, if we react on the same liquid with sodic hydride in solution in sodium amalgam, we obtain an increase in bulk with the production of a butyrous mass which remains for two or three days in the ammoniacal solution.

To prepare this sodic hydride, we started from the interesting experiments made by MM. Troost and Hausfeulle, who have observed that on passing a current of pure hydrogen over sodium kept at a temperature of 350°, a hydride, Na₂H, is formed, having a metallic appearance, and comparable to the hydride of palladium. The solubility of the hydrogen in the sodium is reasonable, but in reality the phenomenon is more complex.

If we keep sodium in a glass U-tube at a temperature of 320° by means of a Darcet metallic alloy bath, we notice that eventually, at the same time that the hydrogen is dissolved in the sodium, a transparent crystallised product is formed, which is produced this time by the combination of the sodium and the hydrogen.

This new compound is comparable with hydride of calcium which we have previously described. When finely powdered, it takes fire on contact with the air; it bursts into flame, with a very slight elevation of temperature in chlorine or oxygen gas; it is very hygroscopic, and decomposes with violence on contact with water, giving sodium and hydrogen. This new hydride of sodium can be separated from the excess of metallic sodium by means of liquid ammonia free from moisture.

The alkaline metal is carried off in the form of sodammonium, and the white hydride remains as a residue.

To sum up, from these preliminary experiments we are able to conclude that by reacting with chloride or iodide of ammonium, in solution in anhydrous ammonia, on sodium amalgam, we obtain a metallic mass in which the hydrogen and the ammonia are found in stable combination at -39°.

This metallic mass, through its decomposition at the ordinary temperature in the absence of water, increases its volume thirty times, and gives off 2 volumes of ammonia gas for 1 volume of hydrogen gas.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 14.

THE CALCULATION OF ATOMIC WEIGHTS.

By F. W. CLARKE.

In the ordinary discussion of ratios relative to atomic weights, different results may be obtained by varying the method of computation. This is due to the fact that the ratios as measured are not absolute, but subject to errors, which should be, but are not usually, distributed. In most cases the tendency is to accumulate all errors, accidental or systematic, upon the constant which is last determined, a procedure which is obviously unscientific. Take, for example, the ratio CaF₂:CaSO₄, upon which our knowledge of the atomic weight of fluorine chiefly depends. In the actual measurement certain errors of observation occur; the data are reduced with assumed values for the atomic weights of calcium, sulphur, and oxygen, which are themselves, to a greater or less extent, inexact, and all of these inaccuracies are brought together in the final result of calculation. If oxygen be taken as the standard of atomic weights, and therefore not subject to error, the atomic weight of fluorine will contain the error of the experiment, plus the errors in the atomic weights of calcium and sulphur; and whether these compensate one another or not we have no means of knowing. Moreover, each error exerts its maximum effect, whose extent can hardly be estimated.

It is generally agreed, with but few dissenting voices, that the work of Stas upon some of the more fundamental atomic weights, is as accurate as the present resources of experimental chemistry can make it. And yet, even here, discrepancies exist which the ordinary method of calculation fails to reconcile. The ratios measured by Stas have been discussed by various writers, especially by Stas himself, by Ostwald, by Thomsen, and by Van der Plaats, and the results of the several computations vary with the method of calculation. Van der Plaats (*Comptes Rendus*, cxvi., 1362), in particular, has illustrated this fact by making two calculations, giving the ratios equal weight in one (A), and weight inversely proportional to the squares of their probable errors in the other (B). In the following table the various results of the reductions appear:—

	Stas.	Ostwald.	Thomsen.	Van der Plaats (A).	Van der Plaats (B).
Ag ..	107.930	107.9376	107.9299	107.9202	107.9244
Cl ..	35.457	35.4529	35.4494	35.4516	35.4565
Br ..	79.952	79.9628	79.9510	79.9407	79.9548
I ..	126.850	126.8640	126.8556	126.8445	126.8494
Na ..	23.043	23.0575	23.0543	23.0453	23.0443
K ..	39.137	39.1361	39.1507	39.1414	39.1403
N ..	14.044	14.0410	14.0366	14.0421	14.0519
S ..	32.074	32.0626	32.0606	32.0576	32.0590
Li ..	7.022	7.0303	7.0307	7.0273	7.0235
Pb ..	206.926	206.911	206.9042	206.9089	206.9308

The differences thus shown are slight, but in the case of bromine they amount to more than two in the second decimal place, an uncertainty greater than is commonly recognised. It is an uncertainty which affects all other determinations depending upon the atomic weight of bromine as an antecedent factor, and which ought to be appreciably diminished. With sulphur and nitrogen the divergences are proportionately even greater.

Now, bearing in mind that the so-called determination of atomic weights is nothing more than the accurate measurement of chemical ratios, let us consider how the latter are to be treated. In the case first cited, the ratio between calcium fluoride and calcium sulphate, the usual method of calculation, is evidently defective, and yet, with existing data, it is the only practicable mode of procedure. With adequate data the ratio should contribute to our knowledge of at least three atomic weights, those of fluorine, calcium, and sulphur, its errors of observation being divided into three parts and not concentrated upon a single factor. That is, the ratio should be combined with other ratios in such a manner that several atomic

weights might be simultaneously computed, all errors being so distributed as to reduce their influence to a minimum. With some groups of ratios calculations of this sort are already possible, and Van der Plaats's reduction of Stas's determinations is a good illustration of my meaning. In this case 23 ratios, involving the atomic weights of 10 elements, were combined into one group of normal equations, and the latter were then solved once for all. The method followed was the well-known method of least squares, and that is the only process which satisfies all conditions. Given enough data of sufficient accuracy, and the method of least squares is applicable to the problem in question; with insufficient evidence or doubtful measurements no solution is satisfactory. The mathematical operations are well understood; it is for the chemist to furnish suitable material to which they may be applied.

In order to illustrate more fully the foregoing considerations, I have combined thirty ratios, involving the atomic weights of silver, chlorine, bromine, iodine, nitrogen, sodium, and potassium, into seven normal equations, in which the constants named appear as the unknown quantities. The ratios are given on pages 53 and 70 of my "Re-calculation of the Atomic Weights" (Smithsonian "Constants of Nature," 1897, Part V.), and contain, in addition to the work of Stas, determinations by Berzelius, Penny, Pelouze, Marignac, Dumas, Gerhardt, Maumené, Turner, Millon, Huntington, Richards, Ramsay and Aston, Hardin, Thomsen, and Hibbs. In short, all determinations of the ratios in question, made up to 1897, are included in the discussion, in order that the effect of such a combination may distinctly appear. The basis of calculation, the standards of reference, are $O = 16$ and $H = 1.0079$, these values being taken as known. Personally, I prefer the hydrogen unit as a starting-point, but in the present case the results are to be compared with those of other computers, as previously cited.

To show the method of calculation, let me first take one or two ratios separately. For instance, the ratio $Ag:Br::100:74.080$ may be written $100\ Br = 74.080\ Ag$, when the symbols Br and Ag represent the two atomic weights respectively. So, also, the ratio $KClO_3:O_3::100:39.154$ becomes in linear form $39.154\ K + 39.154\ Cl = 2920.608$. From thirty equations of this kind the seven normals are constructed, each ratio contributing toward the establishment of each atomic weight represented in it. In this way errors of measurement are divided up and distributed, and even systematic errors find their influence diminished.

If all of the 30 ratios were equal in value and importance, the formation of the normals would be a comparatively simple matter, but such is not the case. Some are very exact, others are more than doubtful, and weight must be assigned accordingly. The rigorous rule is to give each measurement weight inversely proportional to the square of its probable error, and as the latter is given in every case, the rule has been applied. Only, instead of multiplying each equation by its weight, which would give coefficients of excessive size, I have divided them by suitable factors, and so reduced the coefficients to manageable dimensions. The principle is unchanged, but its mode of application is somewhat unusual. The same result, however, is reached in the end, and the element of personal judgment is entirely eliminated from the discussion. Each ratio weighs itself, regardless of any preferences upon my part.

Now, taking the regular method of least squares, the thirty equations, after weighting, reduce to the following normals, seven equations containing the seven unknown quantities:—

1. $+5251.3245\ Ag - 6726.5755\ Cl - 599.0278\ Br - 143.1342\ I - 1238.429\ N - 1871.5887\ Na - 4417.7167\ K = 28800.227$
2. $-6726.5755\ Ag + 12014.6535\ Cl - 24.7694\ Br - 28347.1 + 471.803\ N + 3146.9379\ Na + 6758.0443\ K = 41302.978$

3. $-599.0278\ Ag - 24.7694\ Cl + 716.8553\ Br + 156.250\ N + 204.082\ Na + 27.72\ K = -230.712$
4. $-143.1342\ Ag - 28347.1 + 120.2642\ I + 0.5536\ K = 868.704$
5. $-1238.429\ Ag + 471.803\ Cl + 156.250\ Br + 2954.376\ N - 35.1434\ Na - 334.7707\ K = -76872.85$
6. $+1871.5887\ Ag + 3146.9379\ Cl + 204.082\ Br - 35.1434\ N + 3319.6292\ Na = 1919.906$
7. $-4417.7167\ Ag + 6758.0443\ Cl + 27.72\ Br + 0.5536\ I - 334.7707\ N + 6748.573\ K = 24505.141$

A glance at these equations is enough to indicate some of their deficiencies. The first two only are complete; in the others eight terms are missing. In the fourth equation, which represents iodine, the terms corresponding to sodium, bromine, and nitrogen are absent, and even potassium is represented by a coefficient which is ridiculously small. Many other coefficients are smaller than they should be, and this indicates the inadequacy of some of the data. To be really satisfactory, no one of the forty-nine coefficients should be wanting, and all should be reasonably large. Moreover, every coefficient should represent data drawn from more than one ratio, and in too many instances this is not the case. Were Stas's data alone considered, the normals would be still more incomplete, even though the outcome of the solution might be considered better.

Upon solving the seven normal equations the following values are obtained:—

Ag 107.9525.			
Cl	35.4521	N	14.0527
Br	79.9687	Na	23.0636
I	126.8663	K	39.1573

These, except in the case of chlorine, are considerably higher than any of the values given in the table of reductions derived from the work of Stas, and yet they are not so inconsistent with the latter as they might appear to be at first sight. From the new atomic weights we can compute the value of each one of Stas's ratios, and so compare the result with the measurements which he actually made. This is done in the following table, which gives in each case the lowest and highest determination by the great Belgian:—

Ratio.	Stas.		Calculated.
	Lowest.	Highest.	
$KClO_3:O_3$	39.1527	39.162	39.149
$AgClO_3:O_3$	25.078	25.081	25.078
$AgBrO_3:O_3$	20.349	20.351	20.346
$AgI_2O_5:O_3$	16.972	16.9761	16.972
$Ag:Cl$	32.841	32.849	32.840
$Ag:Br$	74.079	74.083	74.078
$Ag:I$	117.529	117.543	117.520
$Ag:KCl$	69.099 (a)	69.1249	69.114
$Ag:KBr$	110.332	110.361	110.354
$Ag:NaCl$	54.2054	54.2093	54.205
$Ag:NaBr$	95.4383	95.4426	95.440
$Ag:NH_4Cl$	49.592	49.602	49.592
$Ag:NH_4Br$	90.823	90.8317	90.830
$Ag:AgNO_3$	157.463	157.510	157.481
$AgNO_3:KCl$	43.864	43.885	43.886
$KCl:KNO_3$	135.638	135.655	135.654
$NaCl:NaNO_3$	145.443	145.469	145.458

(a) Early work, supplanted by later and better measurements.

In ten cases out of the seventeen the calculated ratios fall within the limits of variation shown by the experiments of Stas; in six cases they are lower than his lowest, and in only one case is the result too high. In no case is the divergence large, and in most instances it is remarkably small. My conclusion from all of these considerations is, that although the new figures should not replace the accepted values for the several atomic weights, they are not to be lightly disregarded. They serve to exemplify the ultimate method of calculation, and to show that the data need reinforcement by additional determinations, which should complete the normals, and raise the weak coefficients to adequate magnitude. When that is done,

when, say, fifty ratios are available instead of thirty, then will the method of least squares be fully applicable to the problem, and its use will give the most probable results. To this end, ratios based upon analyses of sodium bromate, iodate, and iodide, potassium bromate and iodate, and ammonium iodide, are especially desirable. Other ratios connecting chlorine, bromine, and iodine with one another are also much needed, and should not be difficult to determine. For example, the ratio $\text{AgClO}_3 : \text{AgBr}$ ought to be easy of measurement, and also the reverse ratio $\text{AgBrO}_3 : \text{AgCl}$.

If the oxygen-hydrogen ratio be considered the base line in our system of atomic weights, then the determination of the seven constants now under consideration might be compared to a primary triangulation. For this purpose no accuracy is too high, no refinement of details too laborious. If the atomic weights of carbon and sulphur should be included in the scheme, giving 9 normal equations for solution, the range of possibilities would be much increased, and the results of the discussion could be made more satisfactory. With existing data, forty-four ratios, good and bad, are now available, and their distribution in the normals can be seen in the following table. Here each horizontal line represents the left-hand side of one equation, and each figure indicates the number of ratios which would influence a given coefficient:—

	Ag.	Cl.	Br.	I.	N.	Na.	K.	C.	S.
Ag ..	31	14	6	4	7	3	5	7	3
Cl ..	14	20	1	1	9	5	6	—	1
Br ..	6	1	7	—	1	1	2	—	—
I ..	4	1	—	5	—	—	2	—	—
N ..	7	9	1	—	13	2	3	1	—
Na ..	3	5	1	—	2	7	—	1	1
K ..	5	6	2	2	3	—	10	—	—
C ..	7	—	—	—	1	1	—	11	1
S ..	3	1	—	—	—	1	—	1	4

That is, the normal for silver would be formed from thirty-one ratios containing silver, fourteen containing chlorine, &c. The other equations would all be incomplete, twenty-four terms being absent, and eighteen more being affected only by single ratios. To supply the twenty-four and to improve the eighteen is the experimental part of the problem, and this is not so great a task as it might seem to be at a first glance. For example, suppose that the ratio $\text{AgCNS} : \text{AgI}$ were capable of measurement. Its errors of observation would be divided between five elements, eight missing coefficients would be supplied, and twenty-five coefficients in all would be affected. Merely to fill the gaps in the equations, therefore, is easy; to fill them properly is a more weighty question. To this end, forty or fifty new ratios ought to be carefully determined, giving a system of ratios containing from eighty to a hundred in all. For one chemist so great a labour would be hardly possible, but by co-operation between several workers the task might be performed, and in something like reasonable time. The methods of manipulation, the precautions to be observed, the difficulties to be encountered, are all well known, and that consideration helps to simplify the problem. Stas and Marignac have laid the foundations, and with their recorded experience to guide us we should be able to build the structure higher without excessive difficulty.—*American Chemical Journal*, xxvii., No. 5.

Direct Hydrogenation of Acetylenic Carbides by the Method of Contact.—Paul Sabatier and J. B. Senderens.—M. Moursu, who has lately made important syntheses from the two acetylenic carbides, heptene α and phenylacetylene, placed a quantity of these substances at the authors' disposal. To these they applied their method of hydrogenation. The details of the experiment show that copper enables the first stage of the transformation to take place with ease; that is, the passage into ethylenic carbides, but the second stage—that of transformation into formenic carbides—takes place much more slowly.—*Comptes Rendus*, cxxxv., No. 2.

COMMON ERRORS IN THE DETERMINATION OF SILICA.*

By W. F. HILLEBRAND.

To some it may seem as if a threadbare subject had been reopened in the above title, and that concerning a determination of such common occurrence in both technical and scientific operations little remained to be said or done. But that this is not so the following presentation will, I think, make sufficiently clear. Although not much of what I shall offer is really new, even that which is not will well bear repetition, having escaped the attention which it deserves. It seems to be advisable and necessary that public attention should be from time to time called to important facts which have not impressed themselves sufficiently to become a matter of common knowledge and application.

Regarding certain features of the silica determination I have long entertained doubts, but it is only very recently that convenient opportunity has presented itself for an investigation of these points as well as others, the direct incentive being afforded by an experience of the past summer as an outgrowth of the labours of a Committee of the New York Section of the Society of Chemical Industry having for its object the promotion of uniformity in technical analysis.

Uniform samples of raw cement mixture and of finished cement were sent to a large number of analysts for determination of the commoner constituents by the usual technical methods. The same samples were analysed in greater detail by myself and with the same regard to exactness that is usual in analysing rocks and minerals in the laboratory of the United States Geological Survey. Fourteen of the chemists besides myself returned reports, which showed in most cases a marked lack of agreement. The results for silica† only are here presented in tabular form with one exception, which is omitted for the reason that it does not profess to represent silica only, but rather the residue from evaporation of the limestone with acid, hence containing much undecomposed mineral matter.

TABLE I.—Silica Found by different Analysts in the same Samples of Raw Mixture and Finished Cement.

		Limestone mixture.		Finished cement.
Standard		15'18		21'31
1 ..		15'75		20'90
1a ..		15'37		—
2 ..		14'68		20'92
3 ..		13'92		20'06
4 ..		—		20'00
5 ..		14'18		20'26
6 ..		14'70		20'96
7 ..		12'78		20'84
8 ..		13'97		19'82
9 ..		14'44		20'76
10 ..		13'60		19'18
11 ..		14'64		21'46
12 ..		14'18		20'76
13 ..		14'92		21'56
14 ..		13'56		19'53
Highest		15'75		21'56
Lowest ..		12'78		19'18
Difference		2'87		2'38

* Read at the Philadelphia Meeting of the American Chemical Society. From the *Journal of the American Chemical Society*, vol. xxiv., No. 4.

† The full data, together with copies of the outlines of methods employed, were submitted to me for review and suggestion without knowledge on my part of the names of the analysts. My report was transmitted through the Director of the Geological Survey to the Committee of the New York Section of the Society of Chemical Industry, and its report was rendered at the Section Meeting on December 20, 1901, and published in the *Journ. Soc. Chem. Industry*, January 15, 1902.

Without detailing the various means employed for rendering silica insoluble, it may be said that all the workers strove to bring this about by the usual more or less approved methods of drying at temperatures ranging from that of the steam-bath for a long time to 120° and even higher for a shorter time. Despite their efforts there is not only a wide discordance in results, but, with two exceptions in each series, they are all too low. The actual failure to recover all silica is, however, in the majority of cases greater than the figures indicate, by reason of neglect of blast-ignition, or of correction by hydrofluoric acid, or both.

It is evident, then, that none of the methods in common use for rendering silica insoluble can be at all depended on to effect this result. None of the analysts except No. 1 and myself seems to have employed two evaporations with an intervening filtration. All others made but a single filtration, and in this simple difference lies the main solution of the trouble—a fact pointed out by Alex. Cameron with quantitative demonstrations nearly eight years ago (CHEMICAL NEWS, 1894, lxi., 171), but which has apparently almost escaped the notice of chemists. Indeed, I must admit a certain degree of remissness myself, for I have but an indistinct recollection that it was the reading of Cameron's paper when it first appeared which led me to adopt the practice of double evaporations with intervening filtrations which I have followed for a good many years. What influenced this change had escaped my mind until mention of Cameron's paper in a recent number of the *Journal of the American Chemical Society*, followed by its re-perusal, recalled the matter to recollection.

Other earlier writers, as Lindo, Craig, and Gilbert, have recognised the impossibility of recovering all silica by one evaporation, without, however, recommending a repetition after filtration. Instead, dehydration by hot sulphuric acid is often used in iron and steel works, but its use precludes satisfactory determination of other constituents in the same sample.

Cameron used in most of his experiments 2 grms. of silica, about 98½ per cent pure, fused this with 9 grms. of fusion mixture, dissolved the fused mass in hydrochloric acid, and evaporated in porcelain on the water-bath. The drying was continued on the water-bath in several experiments for some time after all acid fumes had ceased; in others, the dehydration was effected by the blue flame of an Argand burner. He found that, with the lower temperature, four—and even five—evaporations and filtrations were needed to reduce the silica recovered to a negligible quantity, but that with the higher heat three—or even sometimes two—sufficed. Furthermore, he showed by one experiment that a very common practice of evaporating several times to dryness with fresh portions of acid without intervening filtrations did not reduce the silica in the filtrate, and that the presence of aluminum, iron, and calcium was without influence on the results.

My own experiments to test all these points were entirely confirmatory of Cameron's, though less unfavorable as to the number of evaporations needed. This I attribute to the facts that I employed from one-half to one-fourth as much silica as he did, thus conforming more nearly to the ordinary conditions of a silicate analysis, and platinum instead of porcelain evaporating dishes. On the water- or steam-bath drying is not so speedily reached in porcelain as in platinum, and very possibly his later siliceous residues came in large part from the vessel itself.

The material employed in my tests was very nearly pure quartz crystal containing 99.88 per cent silica. Amounts of this lying usually within the limits 0.5 and 1 gm. were fused with 5 grms. sodium carbonate, the fused masses dissolved in hot water, then acidified by hydrochloric acid, and the solutions evaporated in platinum vessels for different lengths of time on a steam-bath. Then the residues were digested for a short time with hydrochloric acid and hot water. Prolonged digestion was unnecessary, as only

sodium chloride with mere traces of other salts had to be extracted.

TABLE II.—Silica Found in Filtrates.

	Weight of quartz.	SiO ₂ in first filtrate.	Per cent.	SiO ₂ in second filtrate.
1.. ..	0.8943	0.0078	0.87	0.0012 (a)
2.. ..	0.8208	0.0321	3.91	0.0006
3.. ..	0.6401	0.0218	3.41	0.0005
4.. ..	0.5738	0.0197	3.43	0.0009 (b)
5.. ..	0.7028			0.0000
6.. ..	0.5931	0.0167	2.82	0.0001
7.. ..	0.7309	0.0211	2.89	0.0001 (c)
8.. ..	0.8495	0.0204	2.40	—
9.. ..	0.7841	0.0142	1.81	?
10.. ..	0.7092	0.0089	1.25	0.0004 (d)
11.. ..	0.7324	0.0147	0.64	0.0005 (e)
12.. ..	2.0000	0.0193	0.96	0.0008 (e)

Remarks.

- (a) Duration of first drying not known.
- (b) Duration of first drying brief, not exceeding one to two hours, and in one case the residue still somewhat moist in parts.
- (c) Duration of first and second drying twenty-one hours each. In 9 the mass was ground to powder as soon as free from visible moisture, without marked improvement in the result.
- (d) Several evaporations with fresh acid before first filtration. Total duration of dryings and evaporations, twenty hours.
- (e) Repeated (4) evaporations with water only before first filtration, twenty hours' drying before third filtration.

Experiment 10 was made in order to test the somewhat prevalent practice of evaporating several times with fresh acid before filtering. The result is an improvement over all but the first of the earlier ones, but still leaves much to be desired.

Experiments 11 and 12, in which repeated evaporations with water only were made, show a still better but by no means satisfactory result. The improvement in these last cases is not surprising, but it is to be expected that in ordinary analysis the foreign matter remaining with the silica would be increased by this treatment with water only instead of acid.

In order to learn, if possible, something about the effect of the large amount of sodium chloride in preventing the dehydration of silica, a soluble gelatinising zeolite (thomsonite with 41.17 per cent silica) was experimented with. The foreign chlorides in solution were here, of course, very much reduced in amount. The results are shown opposite 1, 2, 3 of Table III.

TABLE III.—Showing different Solubilities of the Silica Separated in Gelatinous and Granular Form from Zeolites.

	SiO ₂ in weight of zeolite taken.	SiO ₂ in first filtrate.	Per cent.	Duration of drying.
1.. ..	0.4185	0.0065	1.55	2 hours.
2.. ..	0.4142	0.0048	1.16	21 "
3.. ..	0.8265	0.0072	0.87	21 "
4.. ..	0.5736	0.0020	0.35	Brief.
5.. ..	0.5760	0.0023	0.40	Prolonged.

Since the separation of silica from soluble silicates occurs in two forms, gelatinous and granular, according to the species acted on, it seemed worth while to try a non-gelatinising zeolite (beulandite) containing 57.18 per cent silica. The results appear opposite Nos. 4 and 5 of Table III. Manifestly the instantaneous separation of silica in granular form, without first entering into solution, is far more perfect than when it gelatinises.

We have thus seen how the serious errors in silica determinations shown by Table I. can be avoided by two evaporations with intervening filtrations. In extreme

cases a third evaporation may at times be advisable. Furthermore, that prolonged drying is a useless waste of time, except perhaps after the second evaporation, when, if results like 6, 7, and 8, of Table II. can be depended on, it may be of value in every exact work.

(To be continued).

THE POSSIBLE SIGNIFICANCE OF CHANGING ATOMIC VOLUME.*

By THEODORE WILLIAM RICHARDS.

COMPRESSIBILITY is a universal property of matter. It is so essential an attribute of the experimental universe that it is ascribed even to the imponderable and imaginary ether as well as to "material." The three states of matter are compressible in very varying degrees, dilute gases being compressible to a great extent, highly compressed gases and liquids to a far less extent, and solids to an extent usually even less than liquids. The first case has been studied in great detail, the last two scarcely at all.

Compressibility is simply an evidence of work done upon a system by a given pressure. If the application of considerable pressure in a system causes only a slight change of volume, it is evident that there must be other powerful influences at work. Clearly a clue as to the variation in these influences can be found in the quantitative study of the phenomena.

In all reversible cases which may be studied directly, an increase in pressure is accompanied by an increase of resistance to pressure and a diminution of volume. This depends upon the fundamental idea of equilibrium, and is a special case of the general principle sometimes named after Le Chatelier. Working backwards from this idea, one may infer with regard to any given substance at a given temperature, that it is under the influence of great pressure if its volume-change is unusually small under addition of a given pressure.

There are two conceivable causes of great compression in a substance. The pressure may be applied from the outside, or it may be due to the mutual internal attraction or affinity of the smallest particles of the substance for one another. That is, the substance may be compressed either by an outside pressure or by the intensity of its own cohesion. The first may be typified by highly compressed gases, the second by liquids, whose small compressibility may be taken as evidence of great compression.

In solids one must consider also the directive agency which manifests itself in crystalline form and optical structure. In a few cases the "crystallogenic force" seems to be rather directive than attractive; in other cases it seems to have both properties, for considerable diminution in volume may occur. The presence of the crystal-making force complicates the phenomena and is a considerable stumbling-block in the way of the study of the internal tension of solids.

In view of these facts, it seemed to me possible that the study of compression as manifested by atomic volume under different circumstances, as well as of atomic compressibility, might afford some light as to the affinities at work. The attempt, while only just begun, has not been wholly unsuccessful.

Evidently the liquid is the most suitable state in which to study the effects of molecular and atomic compressibility. It is most suitable because the irregularities in the behaviour of liquids are very great, indicating various internal stresses, and because they are nevertheless not at the mercy of the directive crystal-making tendency which superposes its own influence upon that of cohesion. The great difficulty in the subject lies in the fact that the total compressibility of a substance is usually made up of a

number of parts; the molecular compressibility might be due partly to a diminishing of the so-called "free space" between the molecules, as well as to a diminishing of the distance between the atomic centres. In words free from hypothesis, we may say that the compressibility may be made up of a chemical and a physical compressibility. When one comes to compute from compressibility the probable affinities, one is still more at a loss,—for each affinity is a mutual affair, concerning two specific substances. The immense number of variables thus introduced has discouraged most investigators, and I can find little if any hint of the significance of chemical compressibility in the literature familiar to me.

(The considerations of Nordenskjöld are too seriously complicated by uncertain assumptions to have much value. See Ostwald's "Lehrbuch," 1891, i., 850, for these and similar considerations).

In a case of this kind, one naturally seeks at first cases as simple as possible. A study of the volume changes which take place on mixing liquids reveals at first no apparent regularity. In some cases an expansion occurs, but more usually a contraction; sometimes heat is evolved, and at other times heat is absorbed. One law may, I think, be detected in the midst of the confusion, namely, *similar liquids exhibit less change of volume on mixing than dissimilar ones do*; that is, where the affinity of a substance for itself is not unlike that of the substance for another, no great contraction or expansion occurs on mixing. Thus benzol and toluol when mixed scarcely change in volume at all, while alcohol and water contract considerably. That is just what would be expected if affinity is the cause of contraction.

In order to use such facts it is not necessary to imagine an atomic theory adapted to them. Such a theory is ventured upon at the end of this paper, but the facts are significant without it. One only has to bear in mind that liquid and solid substances resist compression, and hence that when we find them compressed we have reason to believe that pressure has been applied upon them. It is rather a matter of common sense than a hypothetical abstract conception.

In order to present in a clear light the complications involved in the study of even a simple series of cases of chemical compression, the facts concerning the molecular volumes of several metals and their oxides are recorded and discussed below.

Molecular Volumes of Oxides.

Sub- stance.	Weight of metal combined with 16 grms. oxygen.	Density of metal.	Density of oxide.	Space occupied by given weight of metal.	Space occupied by corre- sponding weight of oxide.	Excess of volume of oxide.
2Ag ..	215.86	10.56	7.521	20.55	31.55	+11.00
Hg ..	200.00	13.59	11.136	14.71	19.4	+4.7
Cu ..	63.6	8.95	6.40	7.10	12.4	+5.3
Ni ..	58.7	8.9	6.39	6.60	11.75	+5.15
Cd ..	112.3	8.67	6.5	12.95	19.7	+6.75
Zn ..	65.4	6.9	5.6	9.5	14.5	+5.0
Mg ..	24.36	1.74	3.4	14.0	12.0	-2.0
2Na ..	46.1	0.973	2.80	47.4	22.6	-24.8
2H ..	2.0	0.07	1.00	28.2	18.0	-10.0
Si ..	14.2	2.00	2.30	7.1	13.14	+6.0

In compounds of carbon, according to position 4.7 to 12.0

In liquid oxygen at -119° and 50 atmo-
spheres (sp. gr. = 0.65)... .. O = 24.5 c.c.
In liquid oxygen at -181° and 1 atmo-
sphere O = 14.1 c.c.

While in the first part of this paper no atomic hypothesis is assumed, the words atomic volume, atomic weight, and atomic heat will be used in a purely material sense, as the actual constants pertaining to quantities chemically consistent.

The results recorded in this table are typical of the variety of degrees of contraction which take place when sub-

* From the *Proceedings of the American Academy of Arts and Sciences*, xxxvii., No. 1.

stances combine with oxygen. It is evident that in some cases the product occupies considerably more space than the metal from which it was formed, and that in others (typified by magnesium and sodium above) the oxide occupies considerably less space than the metal. This last remarkable circumstance at once emphasises the absurdity of estimating the atomic volume of an element in a compound by discovering the volume-change which takes place when that element is replaced by another. Oxygen cannot be said to occupy a *minus* quantity of space—the only possible outcome of the false assumption in this particular case. The false method gives fairly consistent results among carbon compounds only because of the great similarity of their composition. This consideration leads to the first law underlying the change of volume in chemical or physical change, namely, *the atomic volume is not a constant, but is dependent upon the environment.* This law was first suggested by Horstmann ("Ostwald's Lehrbuch," 1891, I., 389), but he looked upon it rather as the absence of a law than as the presence of one.

If the affinity of oxygen for the metal were the only variable entering into the figures given above, it is obvious that the total contraction, the difference between the volumes of factors and product, would be at once a comparative measure of the attractive forces which produce the compression. This reasoning of course rests upon the plausible ground that a state of being which resists pressure, such as liquid oxygen or solid metal, may be compressed only by the application of pressure. In this case pressure may be supposed to be applied by the mutual affinity. But unfortunately the case is not so simple.

It is clear that in each case recorded above at least three affinities are concerned; first, the affinity of the metal for itself; second, the affinity of oxygen for itself; and third, affinity of the metal for oxygen. The second of these is constant throughout the series, hence for the present comparison it may be considered as a known quantity. Therefore each change of volume may concern at least two unknown quantities. Hence if it were possible to measure either of the two variable affinities, an approximate idea could be obtained concerning the other from these data concerning atomic and molecular volume.

A slight uncertainty is caused also by the possible varying intensity of the "crystal-making tendency" which determines the structure of solids. The small differences caused by this uncertainty may be seen from the following typical calculation. If solid rather than liquid mercury had been chosen above, the atomic volume of the mercury would have become $\frac{200}{14.1} = 14.2$ instead of

14.7, and the excess of volume of the oxide would have been 5.2 instead of 4.7. These differences are unimportant compared with the larger values under consideration; the precise state of the solids or liquids makes less difference than one would have supposed.

Is there any direct method of determining either the mutual affinity of the two elements or the affinity of the metal for itself?

Countless attempts to measure the former have so continually resulted in failure that many chemists are inclined to deny the existence of chemical affinity. The electro-metric method suggested by Ostwald ("The Chemometer," *Zeit. Phys. Chem.*, 1894, xv., 399) clearly measures one of the ways in which chemical affinity may accomplish work, but it is limited in application and only represents a small fraction of the possibilities. The thermal relations are complicated by well-known thermodynamic irregularities, and would be fully significant only at the imaginary absolute zero.

The direct determination of the affinity of a substance for itself is an easier matter, for many of the properties of a single substance, such as volume, compressibility, tenacity, must be associated with this affinity. Let us seek to study these relationships more closely.

If one could only be sure that all substances, when re-

lieved of their self-affinity, would occupy the same volume, the atomic volume itself would be the simplest and most direct means of comparing this property in different substances. The smaller the actual atomic volume, the greater must be the self-affinity. Such an assumption would at first sight seem to be justified, for those elements which have the largest atomic volumes have the least inclination to remain in the elementary states. Deserting the elementary state means introducing other affinities, however; hence the assumption would be unsafe.

It has been already pointed out that compressibility, if measured over a wide range of pressures, might afford a clue to the extent of compression already existing in any given substance. But the comparison of different substances involves the dangerous assumption that all substances would be alike compressible if freed from self-affinity—an assumption which seems more probable than the last, but which nevertheless must be rejected. A much safer measure of the stress under which a single substance rests is the work which heat is able to do upon it. The changing of a simple substance from t° to $t^\circ + dt^\circ$ centigrade must involve the addition to it of an amount of internal work which is represented by the rise of temperature multiplied by the heat capacity of the substance, or $C dt$. In a simple elementary substance, when this work does not involve the alteration of crystalline form or any other apparent change except increase in size, it seems reasonable to consider no other variables, at least as a working hypothesis. If this is the case, we may write $C dt = P dv$, in which P is the internal stress against which the heat-energy is doing work, C the molecular heat capacity, t temperature, and v volume. The stress against which this work is being done is due only to the internal stress and to atmospheric pressure (which latter may be neglected by comparison with the very large value of the former), hence the stress $= P = \frac{C dt}{dv}$. This can apply

precisely only to infinitesimal changes, because in all probability P will vary with the volume. While it cannot be claimed that the expression just given certainly expresses a single pressure pitted against temperature-work, the expression certainly represents a *resultant* tendency which opposes expansion by heat, and therefore, by inference, opposes all other forms of expansion.* It is the *inward tendency*, the opposite to the driving tendency (Richards, *Proc. Amer. Acad. Arts Sci.*, xxv., 471) or fugacity (Lewis).

While then this stress, represented by the quotient of energy divided by change of volume, can hardly represent anything very definite, it must nevertheless be supposed in a general way to increase when the self-affinity increases. Hence, while giving no certain knowledge, its study may give an indication of affinity.

A typical comparison may be made of the two elements zinc and mercury. They are simple, similar, and yet widely different as to their power of holding oxygen. In each case the atomic contraction on union with oxygen is about the same; If we take as the atomic volume of oxygen the atomic critical volume, the contractions are as follows:— $14.7 + 24.5 - 19.4 = 19.8$, in the case of mercury, and $9.5 + 24.5 - 14.5 = 19.5$, in the case of zinc. If the metals were originally subject to the same internal stress, we should infer from the similarity of contractions that the affinities concerned in the two cases were about equal. This inference is, however, overthrown by other facts. Both elements have about the same atomic heat capacity, hence no internal rearrangement takes place in one which is not approximated in the other. On the other hand, the increase in atomic volume for a rise of 1° of temperature exhibited by one is much greater than that exhibited by the other.

If a grm. atom of one element increases more rapidly in size than the grm. atom of another, it is only reasonable

* All the slight data which we possess upon compressibility seem to run parallel with the coefficients of expansion.

to suppose that the heat energy is finding less opposition in the former case. The coefficient of cubic expansion of mercury is 0.000179 at 0° C. and the heat required to raise a grm. through 1° is 0.139 Joule. With zinc the corresponding numbers are 0.000087 and 0.392.* The respective atomic volumes are 14.7 and 9.5. Substituting these values in the equation we obtain:—

$$P_{Hg} = \frac{(200 \times 0.139)}{(14.7 \times 0.000179)} = 106,000 \text{ megadynes per sq. cm.}$$

$$P_{Zn} = \frac{(65.4 \times 0.392)}{(9.5 \times 0.000087)} = 310,000 \text{ megadynes per sq. cm.}$$

Both these pressures are very large, for a megadyne exerted on a square centimetre a pressure of almost an atmosphere. As has been said, they signify a resultant tendency which resists expansion.

It is interesting to note that these stresses agree in their indications with the comparison of boiling-points and latent heats of evaporation. The boiling-point of mercury is 357° C. and that of zinc about 930° C. The latent heat of evaporation of zinc is not known, but there is no reason for believing that in its case Trouton's rule is broken. Hence the criteria all indicate that zinc is harder to dissociate from itself than mercury is.

A comparison of the energy-quotients of several metals, measured in this way, may be of interest.

Metal (in order of boiling-point).	Boiling-point, 760 m.m.	Heat capacity (mayers per grm.)	Cubic coeff. of expansion.	Energy quotient $P = \frac{Cdt}{at \cdot \text{expn.}}$ megadynes.
		C		
		mol. wt.		
Mercury..	357° C. = 630° A.	0.139	0.00018	106,000
Cadmium..	770° C. = 1043° A.	0.23	0.000093	214,000
Sodium ..	860° C. = 1133° A.	1.21	0.00022	53,700
Zinc ..	930° C. = 1203° A.	0.392	0.000087	310,000
Copper ..	Unknown	0.375	0.000050	672,000
Magnesium	1100° ± = 1400° A.	1.02	0.000081	224,000
Lead ..	1400° ± = 1700° A.	0.126	0.000088	162,000
Silicon ..	Unknown	0.7	0.0000230	755,000
Diamond.	Unknown	0.5	0.0000036	4,900,000

In these figures one may find traces of many properties associated with firmness of structure or intensity of self-affinity. For example, the order of sequence of the energy-quotients agrees essentially with that of tenacity and of hardness. There is some relationship also to boiling-points and melting-points, although here there are more exceptions. "Chemical affinity" is so much affected by electrical relations and by atomic volume that one would expect to find regularity only on comparing similar elements. Such comparison (zinc with cadmium, or carbon with silicon) seems to show that the energy-quotient tends to increase with diminishing atomic weight.

(To be continued).

NOTICES OF BOOKS.

Catalogue of Scientific Papers (1800-1883), Supplementary Volume. Compiled by the Royal Society of London. Volume XII. London: C. J. Clay and Sons. 1902. Pp. 807.

AFTER a careful sifting of a preliminary list of periodicals not previously catalogued it was found that upwards of three hundred and fifty serials remained to be dealt with. A list of these, many of which were by no means easy of access, together with the abbreviated references adopted, is prefixed to this volume. A few modifications have been introduced in the method of compilation, which cause the present volume to vary in some respects from those previously issued. Numerous cross references have been given, so that it is hoped no difficulty will arise in discovering the works of any Russian or other author.

* All figures not otherwise designated were taken from the tables of Landolt and Börnstein, 1894.

The New Volumes of the Encyclopædia Britannica. The Fourth of the New Volumes, being Vol. XXVII. of the Complete Work. London: *The Times*. Edinburgh: Adam and Charles Black. 1902. Pp. 742.

THE fourth of the new volumes commences with an important series of articles on electricity and electrical matters in general. Prof. J. A. Fleming contributes five, viz., "Electrical Conduction," "General Principles," "Lighting," "Electric Current and Electric Units," and "Electro-Magnets"; Mr. W. G. McMillan writes on "Electro-Chemistry and Electro-Metallurgy"; Mr. W. C. D. Whetham on "Electrolytic Conduction"; and Prof. J. J. Thomson on "Electric Discharge and Electric Waves."

Prof. H. L. Callendar contributes an article on "Fusion," and Prof. Lunge a very interesting one on "Gaseous Fuel." Natural gas is of importance in some of the States of North America, but the author considers artificial gas of much more general importance. The principle classes of gaseous fuel, as classified in this article, are coal-gas, coke-oven gas, producer gas, Siemens gas, blast-furnace gases, water-gas, semi-water-gas, of which a modification is the Mond gas, and air-gas, which is produced by forcing air through volatile inflammable liquids.

The article on "Artificial Gems," by Sir William Crookes, is of interest; in it the writer describes the artificial production of diamonds, the largest of which, however, has only reached a diameter of 0.5 m.m. The artificial ruby is also described; this substance was first made in a practical manner by MM. Fremy and Feil in 1877, while in later years, Elsner, De Senarmont, Deville, and Caron and Debray have produced these gems with more or less success. Sir W. Roberts-Austen has also recently produced rubies as a by-product in the manufacture of metallic chromium. Other gems which have been produced by artificial means are the sapphire, the Oriental emerald, the Oriental amethyst, the Oriental topaz, and the zircon.

Metallography, an Introduction to the Study of the Structure of Metals, Chiefly by the Aid of the Microscope. By ARTHUR H. HIGGINS, Head of Metallurgical Department, Birmingham Municipal Technical School. London: Macmillan and Co., Ltd. New York: The Macmillan Company. 1902. Pp. 158.

AS metallography is as yet only in its infancy and is not sufficiently developed to permit of its being placed on a strictly logical scientific basis, the author only claims to have made an attempt to lay the principles of the subject before students and workers who are interested in the properties and applications of metals, and to offer a series of original illustrations which it is hoped will assist in making the meaning clearer.

The introductory chapter gives a brief account of the history and development of metallography, including a statement of the nature of alloys as generally recognised by eminent metallurgists, apart from microscopic considerations. An alloy may be compared to a rock which contains different constituents, all of which may be united in the liquid condition, but separate on cooling, forming two or more alloys of different densities and of different fusibilities. Or many solid alloys, when heated, release certain of their constituents that have different freezing-points from the remainder. In fact, a cooling mass of metals often behaves like water containing suspended matter does in freezing, when the ice first formed rejects the foreign matter; and so the portion of the alloy which first solidifies rejects certain other portions of the constituent metals.

Polishing the metal is a matter of great importance for the exact examination of the structure of metals, and the chapter dealing with this subject should be carefully studied. Etching and tinting are also matters of considerable interest, as it is through this means that the structure of the metals is made visible.

The bulk of the book, beginning with Chapter IV., on the "Structure of Iron," deals with the structure of a large number of metals and alloys, and the letterpress is amplified by 96 excellent microphotographs which are deserving of much praise. A glossary of technical terms is to be found on page 149 and following, while a fairly good index completes the volume.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

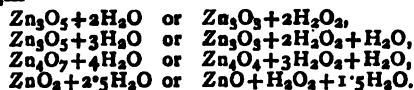
Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 2, July 15, 1902.

Preparation and Properties of a Vanadium Silicide.

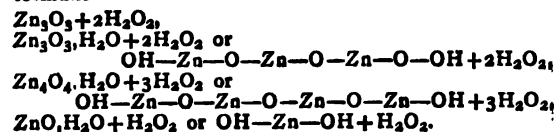
—H. Moissan and M. Holt.—To prepare the compound VSi_2 the authors heated a mixture of vanadic oxide, V_2O_5 , with slightly over five times its weight of pure crystalline silicon in the electric furnace. The reaction takes place according to the equation $2V_2O_5 + 11Si = 4VSi_2 + 3SiO_2$. The same silicide can also be prepared by the reduction of a mixture of silicon and vanadic acid by magnesium powder. Vanadium silicide, as prepared by either of these methods, is in the form of brilliant metallic prisms. It has a density of 4.42, and is fusible and volatile in the electric furnace. An analysis of this substance shows it to have the formula VSi_2 .

Properties and Constitution of Zinc Peroxides.

—M. de Forcrand.—The author, in a previous paper, described four zinc oxides, which, according to their methods of preparation, appeared to be the definite compounds—



After a series of further experiments on these four substances, he believes them to have the constitutional formulae—



The oxides Zn_3O_3, H_2O and Zn_4O_4, H_2O being polyzincic or metazincic acids of the same nature as hydrates of condensed oxides and analogous to the hydrated sulphides Zn_3S_3, H_2O and Zn_4S_4, H_2O . This constitution can be compared to that of perchromic acid, which has the constitution CrO_3, H_2O , according to Moissan, or $Cr_2O_7, 2H_2O, H_2O_2$, according to Berthelot. In all cases the hydrated peroxides of zinc are very different from the hydrated peroxides of calcium, barium, lithium, and sodium.

Oxyisopropylphosphinic Acid.—C. Marie.—The formulae of the salts and the ethers and the existence of a benzoyl derivative justifies the name oxyisopropylphosphinic acid, and gives a means of establishing its formula,

$(CH_3)_2-C-OH$
mula, $\begin{array}{c} | \\ O=P-(OH)_2 \end{array}$. This is the first body of a new series of oxyphosphinic acids which can be compared with the oxyphosphinic acids prepared by the action of PCl_3 on the fatty or aromatic aldehydes.

New Method of Preparation of Substituted β -Ketonic- α -substituted Ethers.—René Locquin.—When the chlorides of the fatty acids act on sodium acetylacetic ethers, a mixture of the two isomeric acyl-derivatives is obtained. The C-acetylacetates, $CH_3-CO-R-CO>CH-CO_2R'$, when treated with aqueous potash or gaseous ammonia, gives a series of acetylacetates, $R-CO-CH-CO_2R'$, possessing properties analogous to those of the acetylacetates, and, amongst other properties, that of replacing one of the hydrogen

atoms of the CH_2 group by a radicle, R' , when treated with a mixture of sodium ethylate and alcoholic iodide, $R'I$.

MISCELLANEOUS.

Institute of Chemistry of Great Britain and Ireland.

—The next Examination in the Branch of Biological Chemistry will be held at the Laboratories of the Institute, 30, Bloomsbury Square, London, W.C., on Tuesday October 21, 1902, and three following days. The Examination will extend over four days, and the syllabus will include Biological Chemistry, with special reference to the Chemistry and Bacteriology of Foods, Water, Sewage, and Effluents, and the practical applications of Biological Chemistry to Industries. Examinations in the Branch of Biological Chemistry of the Final Examination are held annually in October. The Examinations in the other Branches are held in January and July. Candidates intending to enter for the Final Examination in the Branch of Biological Chemistry are recommended to take (after passing the Intermediate Examination, or any Examination qualifying for admission to the Final) a course of study covering the following work:—A course of forty to fifty Lectures on the Morphology and Physiology of Micro-organisms. Practical work to include—(a) Microscopy, the preparation, staining, mounting, drawing, and recognition of specimens; (b) the preparation and study of pure cultures; (c) the conduct of fermentation experiments, and the study of chemical changes brought about by bacteria, moulds, yeast, &c.

New Volumetric Estimation of Bromides in the presence of Chlorides and Iodides.—Von Wesselsky.—Acid chlorine water transforms iodides into iodates and displaces the bromine from bromides, no matter what excess of chlorine is used. In alkaline solution the bromides are transformed into bromates by an excess of chlorine water. On these facts the author has based the following method of estimation:—If the substance does not contain iodine, about 1 grm. of carbonate of potassium and the corresponding quantity of chlorine water are added to the solution; it is then evaporated to dryness. The residue, dissolved in 100–150 c.c. of water, is acidulated and titrated by hyposulphite of soda after the addition of a little iodide of potassium. If the solution contains both bromine and iodine it is placed in a retort, acidulated, and after adding chlorine water in sufficient quantity to oxidise the iodides to iodates and displace the bromine from the bromides, it is distilled in a current of carbonic acid. The bromine and the excess of chlorine are collected in an aqueous solution of 1 grm. of potash; after the potash is completely saturated by the carbonic acid it is evaporated to dryness, and taken up with water and iodide of potassium to titrate, by means of hyposulphite, the iodine displaced by the bromate. Chlorates in small quantities have no action on the iodide of potassium, but iron, arsenic, and antimony ought first to be eliminated in the ordinary way. This method applied to the analysis of mineral waters has given very concordant results.—*Zeit. Anal. Ch.*, vol. xxxix., p. 81.

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NOTICE.

The STUDENTS' NUMBER of the CHEMICAL NEWS will be published on Friday, September 5th. Gentlemen holding official positions in the Universities, Medical Schools, &c., of the United Kingdom, where Chemistry and Physical Science form a part of the education, who have not yet forwarded the necessary information to our Office for publication in that Number, will confer a favour by sending it with the least possible delay.

Advertisements for this Number should reach the Office not later than Wednesday, Sept. 3rd.

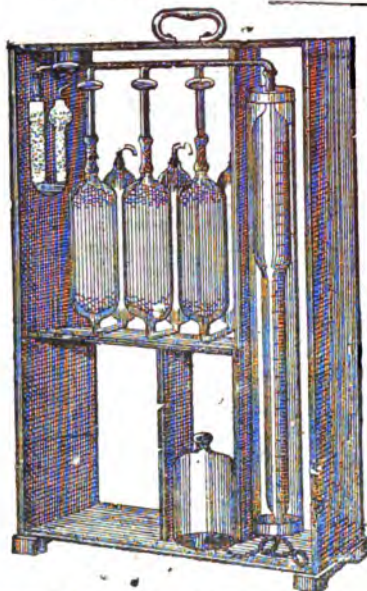
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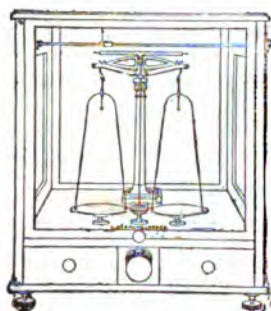
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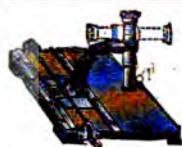
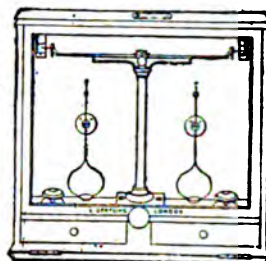


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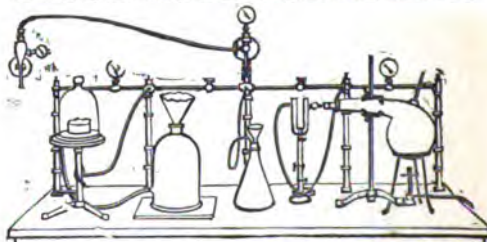
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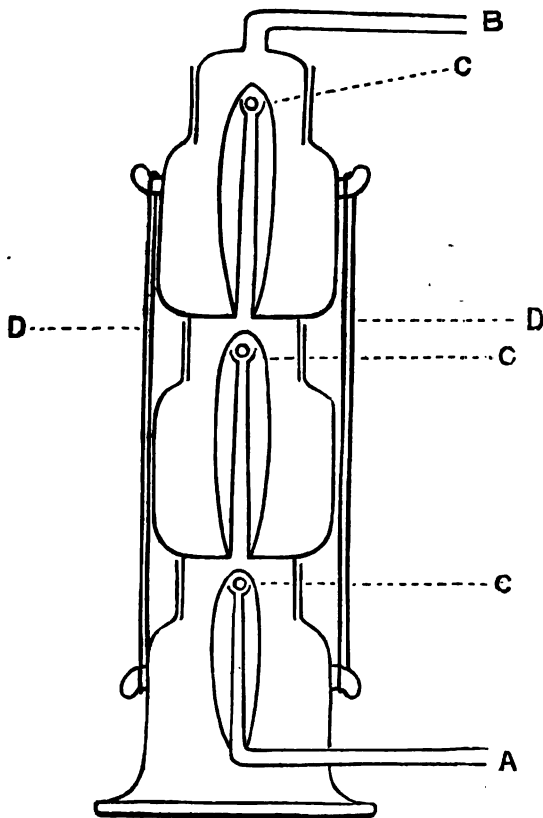
VOL. LXXXVI., No. 2230.

A TRIPLICATE APPARATUS FOR DRYING AND PURIFYING GASES.

By EDWIN DOWZARD, F.C.S.

THE accompanying sketch is that of an apparatus for drying and purifying gases.

The special point about this apparatus is that three different reagents, either solid or liquid, may be used. For example, the bottom cell may contain a strong solution of KHO, the second cell H_2SO_4 , and the third, soda-



A, Inlet; B, outlet; C, C, C, ball valves; D, D, elastic bands connecting top and bottom cells.

(The above sketch is half actual size).

lime. When solids are used, a small quantity of glass-wool should be placed on the floor of cell so as to cover the inlets.

This apparatus is recommended for organic analysis, as it is very efficient, occupies little space, and is easily cleaned and filled. A battery of two triplicate washers is sufficient for all ordinary purposes. The working capacity of each cell is about 45 c.c.

The above apparatus is made by Messrs. Gallenkamp and Co., 19, Sun Street, London,

SOLANUM DULCAMARA.

By FREDERICK DAVIS.

THE *Pharmaceutical Journal* of October 23, 1886, contains the following Editorial Note:—

"Some uncertainty prevails as to whether the fruit of *Solanum dulcamara* is poisonous, and the question having been raised in *Science Gossip* last month Mr. F. Davis has made some experiments on the point, from the results of which it appears that the fruit contains an alkaloid similar to that obtainable from belladonna. Mr. Davis has found that this alkaloid has the power of dilating the pupil of the eye, and he considers that it may be intermediate in character between atropine and physostigmine. The ripe berries yield a larger amount than those which are unripe."

Since the above note appeared I have at various times engaged myself in the further examination of this plant, operating for the most part upon fresh specimens collected upon the Surrey Hills and growing, of course, upon chalk and bed of gravel.

The methods employed were those indicated in Dragendorff's plant analysis.

The substances separated were:—

- | | | | |
|----------------|-----------|------------------|-----------|
| (a) Solanine | } | | Alkaloids |
| (b) Solanidine | | | |
| (c) Solanein | | Glucoside | |
| (d) Dulcamarin | | Bitter principle | |

The solanine was removed from its salts in aqueous solution by amyl alcohol, finally crystallising in four-sided prisms having a melting-point $235^{\circ}C$.

The taste is bitter, with subsequent burning sensation to the tongue and back of the mouth.

The alkaloid gave an alkaline reaction with litmus.

Solanine does not appear to be soluble in water except very sparingly, but the sulphate, hydrochloride, and bimalate are freely soluble; indeed, the alkaloid exists in the ripe fruit as bimalate.

The bulk of the acid of the fruit was proved to be monohydroxy-succinic acid.

The percentage found in the ripe fruits in ten separate determinations varied between 0.3 and 0.7.

Reactions for solanine as found are as follows:—

Phospho-molybdic Acid.—Canary-coloured precipitate.

Potassio-bismuthic Iodide.—Red precipitate.

Froehde's Reagent.—Red, becoming brown, finally yellow.

Concentrated Sulphuric Acid.—Ruddy yellow.

Alcohol and Sulphuric Acid.—Red when warmed.

Does not reduce Fehling's solution.

Solanidine.—This alkaloid was found chiefly in the leaves and young shoots, but it also exists in the fruits; in fact, it would appear that solanidine is the prior alkaloidal product, and that solanine is, as it were, the ultimate product of the plant. Solanidine is soluble in alcohol, whereas solanine is practically insoluble excepting in boiling alcohol of specific gravity 0.840. The salts of solanidine are slightly soluble in water.

Solanidine was removed by chloroform from an alcoholic extract of the plant.

It crystallises in brilliant acicular form, has a bitter and acid taste, and a melting-point $205^{\circ}C$.

Hydroxide of potassium, sodium, or ammonia precipitate solanidine as a gelatinous mass.

Solanein.—Some little trouble was experienced with this substance found in the alcoholic extract with the solanidine, it finally remaining as a non-crystallisable body, horny in character, and of a yellow colour. Solanein is a glucoside, and has a melting-point $208^{\circ}C$. When boiled with very dilute hydrogen sulphate or hydrogen chloride it breaks up into solanidine and grape-sugar. Solanein reduces Fehling's solution.

Dulcamarin.—This substance exists throughout, as evidenced by exhausting the root, stems, leaves, and fruit

separately with acetic ether, then precipitating with basic lead acetate.

It is at first taste intensely bitter, gradually giving place to a sweet and not unpleasant flavour; in fact, exactly the reverse to that which the name would imply. It does not answer the general tests for alkaloids, but dissolves in alkaline hydroxide with a ruddy brown colouration.

Dulcamarin gives a rose-pink colour with concentrated hydrogen sulphate. It is readily soluble in alcohol, and by boiling with dilute hydrogen sulphate is broken up into dulcamaretin and glucose by hydrolysis as follows:—



I find upon referring to the literature upon *Solanum dulcamara* that all authorities speak of dulcamarin, and appear to consider that this bitter principle is the active agent in this plant, some even stating it to be an alkaloid, which, however, it certainly is not, yielding none of the characteristic reactions of these bodies; neither does it form salts with dilute mineral acid. If, however, it be boiled with dilute hydrogen sulphate or hydrogen chloride grape-sugar is produced, and Fehling's solution may be reduced by the results; it is therefore a glucoside as well as a bitter principle.

Some diversity appears to exist respecting the formula of solanine. Firbas gives $\text{C}_{42}\text{H}_{73}\text{NO}_{13}$, whilst Hilger is of opinion $\text{C}_{42}\text{H}_{73}\text{NO}_{13}$ represents it.

Personally, I find the formula to approximate the latter; three separate workings giving by average $\text{C}_{42}\text{H}_{73}\text{NO}_{13}$.

Solanidine is said by Hilger to possess the formula $\text{C}_{25}\text{H}_{41}\text{NO}_2$, Firbas giving $\text{C}_{40}\text{H}_{67}\text{NO}_2$.

Three separate determinations of my own yielded an average $\text{C}_{41}\text{H}_{71}\text{NO}_2$.

Solanine.—Firbas gives the formula of this substance as $\text{C}_{35}\text{H}_{53}\text{NO}_{13}$.

After repeated experiments I cannot agree with this statement, this glucoside giving in my hands $\text{C}_{48}\text{H}_{73}\text{NO}_{13}$. In any case this glucoside contains nitrogen.

After these researches had been completed a sample of solanine was obtained from a well-known German firm and compared with that obtained from the fresh plants as previously indicated; it was found that in taking the melting-point, which approximated 235°C , a sublimate occurred in acicular crystals. Subsequent treatment proved these crystals to be solanidine; it seems, therefore, that commercial solanine is a mixture of solanine and solanidine.

THE DETERMINATION OF COPPER BY ALUMINUM FOIL.*

By GEORGE E. PERKINS.

THE following method is a modification of the process by A. H. Low. The copper is brought into solution as a sulphate by treatment similar to that in Low's modified cyanide process. The solution is evaporated until all nitric acid has been driven off and dense white fumes appear. Water is added until the dilution is about 50 c.c. of water to 20 c.c. of sulphuric acid. Sheet aluminum of about 25 gauge thickness is cut into pieces about 40 m.m. square with one corner of each piece turned up for convenience in handling. Two or three of these pieces are added to the beaker containing the solution of copper. The solution is then boiled. In about five minutes, all of the copper is precipitated upon the aluminum sheets.

Instead of re-dissolving the deposited copper and titrating with cyanide solution, more satisfactory results are obtained by washing the deposited copper into a tared

Gooch crucible, by giving a final wash with alcohol, and by burning off the alcohol and drying. Weigh the result as metallic copper.

In forming the filter, care should be taken that only sufficient asbestos fibre is used to produce a good filter. In washing with alcohol and burning, the same care is needed as in the electrolytic method that too much alcohol is not used.

THE CLASSIFICATION OF THE ELEMENTS.*

By HENRY E. ARMSTRONG, V.P.R.S.

ALTHOUGH no direct evidence acceptable to chemists has been adduced which in any way justifies the belief that the elements are decomposable, it is impossible to resist the conclusion that they are genetically related—so closely in many respects do they resemble a series of related compounds, especially when regarded from the point of view of the organic chemist. The generalisation known as the *Periodic Law* is in itself a justification of this view; the manner in which interrelationship becomes manifest when they are classified in accordance with its canons, being probably the strongest of all the arguments which can be cited as tending to show that the elements are compounds—but compounds very different from those with which we are accustomed to deal. Even in the form in which it was put forward by Mendeleeff, however, the periodic generalisation is but a first approximation; and the great Russian has himself pointed out that it needs improvement and development (Faraday Lecture, *Trans. Chem. Soc.*, 1889, p. 656). As chemists are beginning to recognise this (compare Biltz, *Ber. Dent. Chem. Ges.*, 1892, p. 562), I venture to submit a scheme of classification which I have been led to draw up in writing an article for the Supplement to the "Encyclopædia Britannica." This article, I may say, was sent to press in May, 1900, and the first proof before me is dated November 20, 1900.

The suggestion of an octave of elements having been made in early days, the tendency to adopt an octave system has been irresistible. It is difficult now to discover any real justification of such a practice. As there frequently a difference of (about) a single unit between the atomic weights of contiguous elements, and as the "homologous" elements among those of lower atomic weight differ by (about) sixteen units—I assume that the "elementary difference" may be (about) a single unit, and that the first "horizontal period" comes to an end with oxygen; in other words, that there are sixteen "vertical series" of homologous elements.

In Table I., the elements are entered under whole numbers without regard to their exact atomic weights. The dominant principle on which the arrangement is based is that of maintaining elements which belong to the same family in the appropriate columns. The chemist cannot adopt any other mode of arrangement in view of the uncertainty which attaches to many of the atomic weights.

I have not hesitated to assume that the molecules of argon and similar elements are polyatomic like those of nitrogen—their nearest ally in outward behaviour—and have regarded them as *diatomic*, as the molecules of all the ordinarily gaseous elements are of this order of complexity. It has always been my opinion that they are elements of intense activity, the atoms of which so fully satisfy each other that no residual affinity is manifest in the molecules. From this point of view the appearance of argon immediately after fluorine is quite in order.

Turning to the several periods, the first calls for little remark. Helium is put next to hydrogen—and it may well be that elements somewhat like helium and argon will

* Read before the January meeting of the Rhode Island Section of the American Chemical Society. From the *Journal of the American Chemical Society*, xlv, No. 5.

* A Paper read before the Royal Society, March 20, 1902.

TABLE I.

1 Hg 17 33	2 He 18 34	3 F 19 35 Cl	4 A 20 36	5 21 37	6 22 38	7 Li 23 Na 39 K	8 Mg 24 40 Ca	9 Be 25 41	10 Ne 26 42 Kr	11 B 27 Al 43 Sb	12 O 28 Si 44 45 46 47 48 Ti	13 29	14 N 30	15 P 31	16 O 32 S
53	54	55 Mn	56 Fe 57 58 59 Co Ni	60	61	62	63 Cu 64 65 Zn	66	67 X	68 69 70 Ga	71 72 Ge 73	74	75 As	76	77 Se
78	79	80 Br	81	82	83	84 85 Rb	86 87 Sr	88	89	90 Yt	91 Zr 92 93	94	95	96 Nb	97 Mo
98	99	100	101 Ru 102 Rh 103 Rh 104 105 Pd	106	107	108 Ag	109 110 111 112 Cd	113	114	115 In 116 117 Vc (?)	118 Sn 119 120	121 Sb	122	123	124 Te
125	126	127 I	128	129	130	131 132 133 Os	134 135 136 137 Ba	138 La 139 140 Ce 141 Pr 142 143 144 Nd	(Sm 150, Eu 151, Gd 156, Tb 163, Er 166, Yb 173).						
			191 Os 192 193 Ir 194 195 Pt	196	197	198 Au	199 Hg 200 201	202	203	204 Tl	205 206 207 Pb	208 Bi			240 U

be discovered which will fall into positions 3, 4, and 18; in fact, this region may well turn out to be the nesting-place of such elements. Beryllium is shown separated from lithium and magnesium, but not to an extent which need give rise to objection. It is perhaps not beyond the region of possibility, however, even in the case of an element with so low an atomic weight, that isomorphous mixtures may have passed as pure substances; this possibility has not been sufficiently taken into account in preparing the material for determinations of atomic weight.

In the second period, although it does not come immediately below nitrogen, phosphorus is very near to that element—and sufficiently so to correspond with their very close relationship.

In the third period, when scandium is passed, it is impossible to proceed by units; to bring titanium into position it is necessary to step down five units. Proceeding from titanium, however, vanadium and chromium fall into appropriate positions.

In the fourth period, a similar step down in the fourth column from iron to cobalt and nickel is necessary to bring copper into an appropriate position; and for a like reason zinc is included in a group with copper. There are similar drops in the eleventh and twelfth columns. Selenium appears with 77 instead of 79 as its atomic weight, in order to preserve it in the oxygen-sulphur series; bearing in mind that the atomic weight of sulphur differs from that of chlorine by fully three units, whereas that of selenium is ordinarily supposed to differ from that of bromine by only a single unit, the change does not appear to be an unjustifiable one.

The same argument applies to tellurium in the following period. Personally, I entertain no doubt that the atomic weight of this element will ultimately prove to be considerably lower than that of iodine.

(To be continued).

THE RATIOS OF THE ATOMIC WEIGHTS.*

BY ARTHUR MARSHALL, F.I.C., F.C.S.

MANY remarkable relationships have been noticed between the atomic weights of the elements, but in working them out it has been usual to always refer the atomic weights to that of hydrogen as unity, or that of oxygen as 16. In the following note, after calling attention to some of the relationships that are noticeable under these circumstances, I shall point out some regularities which appear when the atomic weights are referred to entirely different standards.

Since the time of Prout numerous chemists have been struck by the fact that many of the atomic weights approach very closely to whole numbers. This tendency is more pronounced when they are referred to $O = 16$ than when referred to $H = 1$. Strutt, in a recent paper (*Phil. Mag.*, 1901, vi., 311), has calculated the probability of the best known atomic weights approaching as near as they do to whole numbers. Taking from the table published by Richards the nine values given to three decimal places, he has calculated the chances against their approaching so near to whole numbers, using a formula given by Laplace. He finds that the probability is only about 1 in 1000, so that it may be considered practically certain that it is not purely by chance that so many of the numbers bear a comparatively simple ratio to the atomic weight of oxygen.

M. Rudolph (*Chem. Zeit.*, 1901, xxv., 1134) has similarly calculated out the probability figure for the eighteen atomic weights given to two places of decimals in the "didactic" table issued by the German Chemical Society, the values being referred to $H = 1$. It is thus found that the chances are 135 to 1 against the numbers falling out

as they do. I have calculated the probability also for the same eighteen elements using the standard $O = 16$, and I find that here the chances are 4120 to 1 against their approaching so closely to whole numbers. When these atomic weights are referred to $O = 16$ their tendency to approach whole numbers is therefore about thirty times as great as when they are referred to $H = 1$. In the case of an element whose atomic weight is not very high the difference between the two values is but small. The value found for the $H = 1$ table may therefore be due merely to the tendency of the atomic weights to approach whole numbers in the $O = 16$ table. To test this point I excluded the seven atomic weights below 30, and calculated the figure for the other eleven. The probability then rises as high as 1 to 4; that is to say, there is no tendency practically to approach whole numbers in the atomic weights above 30 when referred to $H = 1$. In the case of the $O = 16$ table the probability figure was still only 1 to 37, although all the atomic weights excluded are very near whole integers except that of magnesium.

There is, therefore, a distinct tendency for at least some of the atomic weights to approach whole numbers when $O = 16$. This may be due either to a general inclination of all the atomic weights, or the peculiarity may be confined to a few only, the others being governed by chance. Among the eighteen atomic weights ($O = 16$) there are three which are exactly integers as nearly as can be ascertained—carbon, phosphorus, and cobalt. To these may be added hydrogen (1.0075), the exact value of which is still not quite certain, and may be unity. When these four atomic weights are excluded, the remaining fourteen give a probability figure of 1 : 47. On the other hand, the probability of there being among eighteen numbers four which do not differ from complete integers by more than 0.0075 is only 1 : 7660. From these calculations it appears to be not impossible that the tendency to be whole numbers when $O = 16$ may be confined to quite a few elements.

Prout's hypothesis was definitely disproved by careful atomic weight determinations carried out by various workers, but especially by Stas. But as has been demonstrated, the tendency of certain atomic weights to approach whole numbers cannot be attributed entirely to chance. Although it has been universally the custom to refer all atomic weights either to $H = 1$ or $O = 16$, there is no fundamental reason why this should always be done. The atomic weights were first referred to hydrogen as unity, because that element had, and still has, the lowest atomic weight known. As the ratio between the atomic weight of hydrogen and those of the other elements cannot be determined with great accuracy, it is now more usual to refer the atomic weights to $O = 16$. For practical purposes it is essential that a standard be selected and rigidly adhered to, and the standard $O = 16$ is, on the whole, the most convenient. For the purpose of theory, however, there is no reason why other standards should not be adopted. The choice is large, as the atomic weight of any element may be given any value whatever, and the other atomic weights be then re-calculated from it.

Some three years ago the idea occurred to me to try whether some regularities might not be found between the values which Stas had determined with such accuracy. Taking first the atomic weights of chlorine and bromine, I found that the simplest numbers which expressed their ratio with a sufficient degree of accuracy are 90 : 203. Dividing these numbers by the factor 2.5387, the atomic weights found are 35.451 and 79.962, whereas the figures in the table of the German Committee are 35.45 and 79.96. There is nothing remarkable about this. It was only to be expected that the ratio could be expressed by means of five figures. I next used this factor to multiply the atomic weight of iodine, and I was agreeably surprised to find that it came out very near a whole number. What was still more remarkable was that the same thing occurred with silver. These results are shown in Table I.

* From the *Chemiker Zeitung*, July 19, 1902.

TABLE I.

Element.	Ratio.	Factor.	Atomic wt.	In table of German Com.
Cl ..	90	$\div 2.53868 =$	35.451	35.45
Br ..	203		79.903	79.96
Ag ..	274		107.930	107.93
I ..	322		126.838	126.85

These results can hardly be due to simple chance. I hesitate, however, to apply the theory of probabilities in this case, because I have myself selected the elements dealt with. Fluorine apparently does not fit into this scheme, nor does cyanogen. Several other elements, however, give figures approaching whole numbers when multiplied by the factor, but this is probably due entirely to chance.

The researches of Stas dealt not only with the above four elements, but also with the alkali metals. Mere inspection of the figures in this case shows that values very near can be obtained by multiplying whole numbers by the factor 1.004.

TABLE II.

Element	Ratio.	Factor.	Atomic wt.	In table of German Com.
Li ..	7	$\times 1.004 =$	7.028	7.03
NH ₄ ..	18		18.072	18.07
Na ..	23		23.092	23.05
K ..	39		39.156	39.15
Rb ..	85		85.34	85.4

Except in the case of sodium the values are identical, and even in that case the difference is not great. Other elements whose atomic weights give practically whole numbers when divided by 1.004 are:—Pb, Cd, V, Pt, Sn, Ca, Al. In practically none of these cases, however, are the atomic weights known with any great accuracy, and as most of the elements in question are distributed through the Periodic Table in apparently haphazard manner I prefer to ascribe these results to mere chance.

In spite of the enormous amount of time, skill, and ingenuity which have been devoted to the determination of the atomic weights, the degree of accuracy with which the majority of them are known is too small to allow of this investigation being carried very far. Nevertheless, a very remarkable regularity appears in the horizontal series of elements from vanadium to zinc inclusive, as is shown in Table III.

TABLE III.

Element.	Ratio.	Factor.	Atomic wt.	In table of German Com.
V ..	54	$\div 1.0551 =$	51.18	51.2
Cr ..	55		52.13	52.1
Mn ..	58		54.97	55.0
Fe ..	59		55.92	55.9
Ni ..	62		58.76	58.7
Co ..	—		—	59.00
Cu ..	67		63.50	63.6
Zn ..	69		65.40	65.4

Here, again, the agreement is absolute except in the case of copper, where the difference only amounts to 0.1. It may be mentioned that Ostwald gives the atomic weight of copper as 63.44. Cobalt does not fit in with the other elements at all, but the researches of T. W. Richards tend to show that the ratio O : Co = 16 : 59 exactly.

The facts here pointed out are remarkable, and so far as I know have not been observed before. There are evidently some unexplained regularities in the ratios of the atomic weights. As to the theoretical significance of these regularities, in the present state of our knowledge it appears somewhat premature to discuss it, but there is evidently a connection between the regularities and the phenomena of isomorphism.

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COMMON ERRORS IN THE DETERMINATION OF SILICA.*

By W. F. HILLEBRAND.

(Concluded from p. 81).

Causes of the Solubility of Silica under the Conditions Given.

IN explaining the partial solubility of silica after drying at temperatures much above that of the water-bath it has been customary to assume the formation of soluble silicates by action between some of the silica and salts present. This is an undoubted cause, and one which should become more active with increasing temperature, though Gilbert's work does not seem to indicate this except when magnesium is present in quantity. But, if true at 120°, it must probably still be true to some extent at 100°.

Another explanation for the incomplete dehydration at 100° is assumed to be the protecting influence of other salts, and to remedy this, repeated additions of acid—or, perhaps better still, of water—are sometimes resorted to (see Experiments 10—12 of Table II.).

Again, it may be that the silica separated is itself soluble enough in acid to cause an appreciable error. The experiments reported below show clearly what the action of hydrochloric acid is on silica which had been obtained by fusing quartz with sodium carbonate and drying the evaporated hydrochloric solution for twenty-one hours on the steam-bath. This silica (about 0.65 gm.) was first extracted with a little acid, then thoroughly washed with hot water, rinsed back into the dish, and digested with hydrochloric acid of about 1.10 sp. gr. under varying conditions.

- Digested twenty-five minutes on the steam-bath with about 25 c.c. acid. Silica in filtrate 0.0029 gm.
- Same silica further treated as in *a* for one and one-half hours. Silica in filtrate 0.0026 gm.
- Same silica gently boiled in platinum for thirty minutes with above acid. Silica in filtrate 0.0022 gm.

There was thus extracted with ease nearly 1.2 per cent of the total silica without counting the probably larger amount held by the original filtrate, and it is to be presumed that the effect of the boiling would have been greater had it preceded instead of followed the quiet digestion.

It is thus plain how a portion of the silica always found in the filtrates gets there and that it is hopeless to try to prevent this by a single prolonged drying. Once the bulk of the silica is removed by filtration, however, then, after a second evaporation, because of its tendency to collect in clots of sensible size and consequent small surface exposure, the amount of silica going into solution is very small, and by no means in proportion to that of the acid used.

Determination of Silica that has Escaped Separation by the Usual Processes of Dehydration.

I pass now to another phase of the determination of silica, which, so far as I am aware, has never been investigated.

While it has long been known that some silica passed into the filtrate in silicate work, it was supposed to be recovered in the ordinary course of analysis with the ammonia or basic acetate precipitate, whence after ignition and weighing it could be obtained in insoluble form by fusing the mixture with potassium pyrosulphate. I purpose showing that these suppositions are both in part erroneous, that the residual silica is, as Lindo says, not wholly precipitated by ammonia, and that the ordinary treatment of

* Read at the Philadelphia Meeting of the American Chemical Society. From the *Journal of the American Chemical Society*, vol. xiv., No. 4.

the pyrosulphate fusion mass recovers but a minor part of that which was thrown down with the oxides of iron and aluminum.*

The experiments of Table IV. were made with a hydrochloric solution containing 0.0101 grm. of silica in 10 c.c. and with a solution of aluminum chloride free from silica. The amounts used were chosen to represent approximately the conditions most frequently prevailing in silicate analysis after but a single evaporation to separate silica. Precipitation was effected by ammonia at boiling temperature in a solution of about 300 to 400 c.c. volume containing 25 c.c. of hydrochloric acid of 1.10 sp. gr. As soon as settled, the alumina was filtered, washed, ignited, and fused with potassium pyrosulphate free from silica. The fused mass was generally dissolved in hot water acidulated with sulphuric acid, and once in warm water only, and the residual silica was collected, weighed, and corrected by hydrofluoric acid. In two cases (8 and 9) the filtrates were evaporated with excess of sulphuric acid, heated till fumes came off in quantity, and the separated silica collected after cooling and dilution of the solution. The filtrates from the alumina were evaporated, ignited to remove ammonium chloride, and the silica recovered by evaporation with a few drops of hydrochloric acid and further treatment as usual.

TABLE IV.—Silica in Alumina and in Filtrate from Alumina after One Precipitation by Ammonia.

	Al ₂ O ₃ used.	SiO ₂ used.	SiO ₂ in filtrate.	SiO ₂ recovered from Al ₂ O ₃ solution.	SiO ₂ recovered from K ₂ S ₂ O ₇
1.	0.19	none	none	none	—
2.	0.19	0.0101	—	0.003	—
3.	0.19	0.0101	—	0.0037	—
4.	0.19	0.0101	0.0020	—	—
5.	0.19	0.0101	0.0021	—	—
6.	0.19	0.0101	0.0007	0.0034	—
7.	0.19	0.0101	0.0019	0.0021	—
8.	0.20 (a)	0.0101	0.0007	0.0033	0.0060
9.	0.19	0.0101	0.0021	0.0023	0.0058

(a) Fe₂O₃.

Treatment of about 0.01 grm. of ignited precipitated silica with fused pyrosulphate resulted in the solution of 2 m.grms. of it and of 2.2 m.grms. in another test. Not being evenly distributed through a mass of other oxides undergoing solution, the solvent effect of the pyrosulphate in these cases may reasonably be supposed to be less than in the tests of the table.

We see here how in practically all ordinary silicate analyses a portion of the silica, sometimes very small, escapes being weighed at all, since it mostly passes on into the filtrate from the magnesia. When a double precipitation by ammonia or sodium acetate is made, instead of one as in the above tests, the loss will be greater than the table shows.

We further see how, when correction is made for the silica with the alumina, it is only in small part recovered and the alumina is consequently made to appear too high when determined indirectly. Here we have an added argument in favour of endeavouring to collect all silica at the outset, for it is shown that the expectation of recovering the whole of a missing portion later is based on erroneous assumptions. Therefore Gilbert's conclusion, because he found very little silica with the alumina after fusion with

pyrosulphate, that there is "no tendency for silica to recombine with the lime and alumina" (in non-magnetic silicates) "even at a temperature of 280° C." (*Technology Quarterly*, 1890, iii., 63), is perhaps not well founded. Since his further conclusion that silica is almost completely dehydrated at 120° (*loc. cit.*, p. 64) is based on this same reason, his analytical results lose much of their value.

As showing the errors in the silica results that may be incurred in the analysis of certain siliceous ores where potassium pyrosulphate is employed as the means for breaking them up, I will here give the figures for two titaniferous magnetites. The lower values give the silica obtained in the usual way from the mixture of silica and silicates left after fusion with pyrosulphate, and the higher those afforded by direct fusion of the ores with sodium carbonate.

	I.	II.
Silica	{ 11.73 12.42	{ 20.82 21.42

Complete analysis of the ores showed such a deficiency when pyrosulphate was used that the cause was sought, and found to be as stated above. It was this observation which led to a portion of the work summarised in Table IV.

The Proper Temperature for Ignition of Silica.

Most authorities, Fresenius included, have directed that blast temperatures be used in order to obtain the correct weight of ignited silica. It is only recently that doubt has arisen as to this because of the experiments of Lunge and Millberg (*Zeit. Angew. Chem.*, 1897, p. 425), who, employing silica derived from silicon tetrafluoride, observed no further loss in weight over the blast after sufficient exposure to the full flame of a good Bunsen lamp. This was in direct conflict with all my experience in silicate work, and as a result of friendly correspondence Professor Lunge caused the matter to be re-investigated in his laboratory a year ago, and sent me the following table of results, which I take the liberty of making public:—

TABLE V.—Loss in Weight of Pure Silica from SiF₄ at different Temperatures, as Determined by Dr. Lohföser, in Professor Lunge's Laboratory.

Temperature.	Time. Minutes.	a.	b.	c.
105°	—	0.1840	0.1778	0.2020
Dark redness	30	0.1643	0.1584	0.1803
"	30	0.1640	0.1584	0.1800
Full flame of burner ..	30	0.1622	0.1566	0.1779
"	30	0.1620	0.1564	0.1778
Blast	30	0.1619	0.1564	0.1776

It is but natural that Professor Lunge should write:—"From these results I must conclude that Millberg and myself were right."

The following two tests by myself on silica similarly obtained confirm the above, and show, after blasting, further slight losses comparable with those appearing in Professor Lunge's table and of the same order as those produced by more than half-an-hour's blasting with silica precipitated by acid (see Table VIII.).

TABLE VI.—Showing Loss in Weight of Pure Silica from SiF₄, according to Tests made by Myself.

	a.
Full burner flame, thirty minutes	0.5053
Blast flame, thirty minutes	0.5017
	0.5014
	b.
Full burner flame, forty-five minutes	0.5030
Full flame, forty-five minutes	0.4997
Blast flame, forty minutes	0.4995
	0.4991 (a)

(a) This loss is partly chargeable to the crucible itself.

* Lindo (*CHEMICAL NEWS*, 1889, ix., 14) gives no quantitative data, and in all his experiments (with glass) the ammonia precipitates were, of course, very small. It is fair to conclude from his addition of ferric chloride to the filtrates from them in order to recover by its precipitation by ammonia the "last trace" (*loc. cit.*, p. 40) of silica, that he would not have suspected the presence of any silica in these filtrates when analysing mixtures containing considerable aluminum or iron oxide. Lindo's observations are of value, but his analytical results are not, for the reason that he operated largely in glass vessels and his alkaline solutions remained long in contact with glass, hence must have taken silica from them, notwithstanding which his analyses show without exception a large loss.

These slight losses are not due to mechanical carrying off of silica, for the latter had utterly lost its extraordinarily down-like character during the early heating and become converted by the blasting into a coherent cake of enormously reduced bulk.

Let me now, however, present another series of results furnished by silica otherwise precipitated. The silica used was obtained by three evaporations from the quartz that served for the earlier experiments of this paper, and the initial weights are those found after an exposure of about one hour to the full Bunsen flame. The weights given represent the silica as corrected by hydrofluoric and sulphuric acids for the few tenths of a milligram of non-volatile salts always present. The crucibles were found not to lose weight themselves over the blast. The quartz powder employed was 99.88 per cent pure, as found by careful evaporation with hydrofluoric and sulphuric acids.

TABLE VII.—Showing Differences in Weights of Silica Precipitated from Sodium Silicate when Ignited over Bunsen Burner and Blast-lamp.

	Silica found.		Loss in weight.	Percentages found.	
	Burner, one hour.	Blast, half hour.		Burner.	Blast.
1. 0.5738	0.5761	0.5735	0.0026	100.40	99.95
2. 0.5931	0.5945	0.5930	0.0015	100.24	99.98
3. 0.6401	0.6450	0.6394	0.0056	100.76	99.90
4. 0.6638	0.6668	0.6628	0.0040	100.45	99.85
5. 0.7028	0.7058 (a)	—	—	100.25	—
6. 0.7309	0.7342	0.7306	0.0036	100.45	99.96
7. 0.8208	0.8271	0.8206	0.0065	100.77	99.98
8. 0.8495	0.8521	0.8484	0.0037	100.31	99.88
9. 0.8943	0.8996	0.8936	0.0060	100.59	99.92
10. —	0.9989	0.9989	0.0091	—	—

(a) Two hours.

Results similar to the above are repeated in every silicate analysis that is made in our laboratory, and others have told me their experience is the same. It is as clear as daylight that the blast is a necessity if the work is to be at all accurate. In every one of the above instances the percentage found without blast is far in excess of 99.88—the true value for silica in the quartz,—and is as invariably brought very near this value, and with far smaller variations from a mean, when the blast is applied (see below). The above values for "burner" percentages very well represent the usual lack of exact agreement in series of duplicate silica determinations when the blast has not been used (see Gilbert's paper), while those of the next column show of what accuracy the determination is susceptible when all the conditions of success are understood and applied.

From the foregoing it appears that the silica separated by water from silicon tetrafluoride, and that by hydrochloric acid from sodium silicate, are in different conditions and behave differently when strongly heated; that Professor Lunge's conclusion that silica need not be blasted, while correct for one form of silica, cannot be applied, as he supposed, in analytical work.

I have always, until lately, regarded one-half hour's blasting as sufficient in all cases for as much as a gram of silica, but that this length of time is often insufficient to secure constant weight is indicated by some of the results in Tables V. and VI., and still more by those in Table VIII.

TABLE VIII.—Showing Need of very long Blasting at Times.

Heat.	Time in minutes.	Weights of silica.					
		1.	2.	3.	4.	5.	6.
Burner	60	0.6691	0.7041	0.9989	—	0.4175	0.4220
Blast	30	0.6657	0.6982	0.9905	0.7208	0.4154	0.4192
"	30	0.6656	0.6979	0.9902	0.7201	—	0.4187
"	30	0.6651	—	0.9899	0.7193	0.4152	0.4185
"	30	—	—	0.9897	0.7194	—	—
"	30	—	—	—	0.7194	—	—

The progressive losses shown here are not chargeable to the crucibles, nor were they due to mechanical loss of silica or to volatilisation of included salts, and they explain very well the fact that nearly all the figures of the first column in Table V. slightly exceed the true value for silica in the quantity used, for they were obtained by blasting for only one-half hour. They may also serve to explain in small part the excessive summations often encountered in rock analyses. These later losses can exert little effect when the silica percentages are small, but when large, as in rock analysis, they may at times be of moment and necessitate blasting for more than half-an-hour.

In the foregoing I have entered into such detail as the exact scientific worker needs for his enlightenment, and have carried my separations and ignitions farther than the technical chemist ordinarily cares to or can proceed. But in its main features, what I have said concerns him not less than the research chemist, and I trust that in some respects it may help to ease his path.

Summary.

Statements of earlier writers are fully confirmed, that silica cannot be rendered wholly insoluble by a single or any number of evaporations with hydrochloric acid when followed by a single filtration, no matter what temperature may be employed, but that two or more evaporations alternating with filtrations are necessary to secure satisfactory results.

It is shown that the generally accepted view that any silica passing into the filtrate is wholly thrown down by ammonia or sodium acetate in presence of much aluminum or iron is incorrect. Also that silica is appreciably soluble in melted potassium pyrosulphate, and that, consequently, when siliceous oxides of iron and aluminum obtained in analysis are then fused, their silica contents are only in small part left undissolved when the fused mass is taken up with water or acid. Both these sources of error are avoided by separating all silica at the start as above.

The need of blast ignition in order to get the correct weight of silica obtained in analysis is proved. The opposite conclusion of Lunge and Millberg, being based on what seems to be a different behaviour of the silica derived from silicon tetrafluoride, is therefore not justified.

THE POSSIBLE SIGNIFICANCE OF CHANGING ATOMIC VOLUME.*

By THEODORE WILLIAM RICHARDS.

(Concluded from p. 83).

HAVING thus plausible inference, from independent sources, as to the relative values of the compressing agencies existing in metals at the ordinary temperature, it is worth while to study the correction which must be applied to the volume change exhibited in chemical combination with another element. In zinc the self-affinity is so great (boiling-point = 1200° A.) and the metal is hence already so compressed, that a given further pressure causes less change in its volume than it would cause in the case of mercury. That is, the mercury contracts more than zinc when it is oxidised. Hence the difference between the volume of the oxide and the volume of the metal gives too low a value for the volume of the combined oxygen in the case of mercury. Thus the contraction of the oxygen is really less in the case of mercuric oxide, although it appears to be the same.

Without going further, one can explain by means of these considerations the behaviour of zinc and mercuric oxides, when subjected to high temperatures. The 16 gms. of oxygen in mercuric oxide occupies a larger space than an equal weight in the case of zinc, hence one infers

* From the Proceedings of the American Academy of Arts and Sciences, xxxvii., No. 1.

that it is less compressed by its affinity, hence the affinity must be less. This smaller affinity should be more easily overcome by rising temperature, a prediction which agrees with facts. Thus there appears to be in this case a connection between the compression of substances and their tendency to combine one with another.

The case under consideration is typical. In the case of sodium and magnesium, the affinity of the metal for oxygen is so enormous as to overcome easily the large affinity of the metal for itself, and besides this to compress both metal and oxygen together into a space smaller than that previously occupied by the metal. This fact corresponds with the great difficulty of decomposing sodic and magnesian oxides. Metallic magnesium probably has as energy-quotient a stress more than four times as great as sodium (see table on p. 83); hence the total contraction on combination with oxygen is less than in the case of sodium. Comparison with the cases of mercury and zinc will show that this small contraction does not necessarily conflict with the fact that magnesium decomposes sodic oxide at high temperatures. Again, the contraction involved in the formation of argentic oxide is very slight. In this case the large volume of oxygen is not concealed by the contraction of the metallic element, as it was in the case of mercury, for silver is not particularly compressible. Hence one can infer that the affinity of silver for oxygen is smaller than that of magnesium for oxygen—an inference which agrees with fact. Moreover, since the relation is nearly additive, that is, neither silver nor oxygen change much in volume on combination, their combination is easily shifted; that is to say, silver oxide is easily decomposed by heat.

Of course many tables comparing the molecular volumes of solids and liquids might be drawn up, since a very great number of specific gravities have been determined. A table containing chlorides of the metals already considered may be of interest.

Molecular Volumes of Chlorides.

Sub- stance.	Weight of combined metal with 35.5 grms. chlorine.	Density of metal.	Density of chloride.	Volume of given weight or weight of metal.	Volume of corre- sponding chloride.	Excess of volume of chloride above metal.
Ag ..	108 ⁵	10.56	5.53	10.27	45.90	+15.63
$\frac{1}{2}$ Hg ..	100 ⁵	14.00	5.42	7.30	25.5	+18.2
Hg ..	200 ⁵	14.00	7.10	14.00	33.2	+19.2
$\frac{1}{2}$ Cu ..	31.8	8.95	3.05	7.10	25.4	+18.3
$\frac{1}{2}$ Co ..	28.5	9.00	2.94	3.16	21.8	+18.64
$\frac{1}{2}$ Cd ..	56.2	8.67	3.7	6.47	24.8	+18.33
$\frac{1}{2}$ Zn ..	32.7	6.9	2.753	4.75	25.0	+20.25
Mg ..	12.2	1.74	2.177	7.0	21.95	+15.00
Na ..	23.05	0.973	2.15	23.7	27.2	+4.2
K ..	39.14	0.875	1.995	45.7	37.3	-8.4
Rb ..	85.44	1.52	2.21	56.1	55.0	-1.0
H ..	1.01	0.07	1.27	14.1 (?)	28.9	+14.7

Combined with carbon ..	22.8
Liquid chlorine at -80° (boiling-point, 760 m.m.; sp. gr. = 1.66) ..	21.5
Liquid chlorine at +80° (sp. gr. = 1.20) ..	29.6

Here the variations in contraction are less than they were before. Chlorine evidently possesses more equally distributed affinities than oxygen does, and apparently somewhat weaker ones. The two most interesting features of this table, which may be seen without the elimination of the self-affinities of the several metals, are the small excess in the case of silver, and the larger excess in the case of mercurous chloride. This is quite in accord with the facts; for argentic chloride is more stable than the oxide, and mercurous chloride easily splits into mercuric chloride and mercury (Richards, *Proc. Amer. Acad. Arts Sci.*, 1897, xxiii., 9).

The case of the hydroxides is especially interesting.

Molecular Volumes of Hydroxides.

Sub- stance.	Weight of combined metal with 17 grms. hydroxyl.	Density of metal.	Density of hydroxide.	Volume of given weight of metal.	Volume of corre- sponding hydroxide.	Excess of volume of hydroxide above metal.
Ag ..	The hydroxide is exceedingly unstable.					
$\frac{1}{2}$ Hg ..	It is doubtful if the hydroxide exists.					
$\frac{1}{2}$ Cu ..	The hydroxide cannot be dried without decomposition.					
$\frac{1}{2}$ Co ..	28.5	9 ⁵	3.597	3.16	12.67	+9.51
$\frac{1}{2}$ Cd ..	56.2	—	4.79	6.47	15.25	+8.78
$\frac{1}{2}$ Mg ..	12.2	—	2.36	7.0	12.90	+5.90
$\frac{1}{2}$ Sr ..	43.83	2.54	3.62	17.3	17.0	-0.3
Na ..	23.05	0.973	2.13	23.7	18.80	-4.9
K ..	39.14	0.875	2.044	45.7	27.5	-18.2
Hydroxyl in organic compounds ..						+12.0
Hydroxyl in hydrogen dioxide (sp. gr. = 1.50) ..						11.4

The density of the hydroxide of zinc has not been accurately determined; indeed, the data concerning cobalt, cadmium, and magnesium are not very trustworthy on account of the amorphous condition of most hydroxides. It is interesting to note that in this table, where the substances are arranged in the order of the contraction which ensues when hydroxyl combines with the metal, should also be arranged in the electro-chemical order. That is to say, the solution tension of a metal appears to be associated with the excess of affinity of the metal for hydroxyl over its affinity for itself, and intensity of potential seems to be associated with intensity of atomic compression. The inference to be drawn from this comparison is, of course, that the formation of the metallic ion in water is connected with the affinity of the metal for water—an affinity which manifests itself even when both of the "bonds" of oxygen are filled.* Similar attraction for nitrogen or sulphur would explain cases in which the solvent does not contain oxygen.

If this is true, contraction should take place when salts are dissolved in water. This inference is amply verified by facts. In some cases the solution occupies even less space than the water alone, involving a total contraction greater than the volume of the salt itself. The best known of these cases are those of lithic, sodic, and baric hydroxides, and cobalt, nickel, zinc, and magnesium sulphates, but undoubtedly others exist (Thomson, "Thermochemische Untersuchungen," 1882, I., 45; MacGregor, *Trans. Roy. Soc. Canada*, 1890, p. 19; 1891, p. 15; *Trans. Nova Scotia Inst. Nat. Sci.*, 1890, vii., 368). In a large majority of cases when an electrolyte is dissolved in water, the sum of the volumes of salt and of the solvent taken together considerably exceeds the volume of the solution. This contraction is usually ascribed wholly to the dissolved substance in dilute solutions (Van't Hoff, "Vorlesung. Phys. Theoret. Chem.," 1900, III., p. 41; Drude and Nernst, *Zeit. Phys. Chem.*, 1896, xv., 79, ascribe this contraction to "electrostriction"), but it seems to me that the behaviour of the salts named above proves the falsity of this method of calculation. The water as well as the salt must contract when a salt is dissolved. So many complications are concerned in the act of the solution of an electrolyte that it is difficult to unravel the tangled clues; but the wide deviations exhibited by different substances seem to indicate that there are present overlapping contractions and expansions, the resultant of which is a smaller quantity than some of the individual influences. Such contractions and expansions are just what one would expect to find in a readjustment of affinities.

In considering the simpler case of solid non-electrolytes, one usually finds here also a contraction upon solution, although less marked than in the extreme cases named above. For this reason, one is inclined to ascribe the act of solution of all kinds primarily to the affinity of the

* Brühl has suggested that oxygen is the cause of dissociation, but he ascribes it rather to quadrivalence than to a general affinity.

solvent for the dissolved substance. The solution tension of a metal or salt becomes simply a balance or ratio of attractions—the separating tendency of heat upon the dissolving phase is much assisted by the attraction from outside. This is of course no new idea. The possible method of treating mathematically these balanced influences is suggested in a recent paper on the “driving tendency” of reaction (Richards, *Fourn. Phys. Chem.*, 1900, iv., 385; see specially p. 391).

That electrolytic separation also should be assisted by the outside attraction for the solvent is almost a foregone conclusion. This may be inferred from the contraction shown by most electrolytes on dissolving. Hence may arise the various contact-potentials exhibited by the same substance in different solvents; for different solvents must possess different affinities. Hence also one would expect to find a much greater potential needed for the dissociation of gases than for that of dissolved substances.

The mechanism of electrolytic dissociation in gases is now usually explained by the aid of the ingenious hypothesis of “electrons,” as amplified by J. J. Thomson and his students in the brilliant experimental researches published in the recent volumes of the *Philosophical Magazine*. This daring hypothesis must not be accepted without reservation, however. Some physical objections to it have been suggested by Ernest Merritt in his interesting address to the American Association for the Advancement of Science (*Proc. Amer. Assoc. Adv. Sci.*, 1900, p. 49); and other objections arise when one tries with its aid to unravel the tangle of influences involved in purely chemical action. The rejected alternative of imagining the atom as indivisible, but as capable of receiving widely varying electric charges under widely different conditions, has some advantages which the opposite hypothesis does not possess. The subject is much too large for discussion here, however. One phase of it, which bears directly upon the subject of the present paper, may receive brief notice.

The results of Thomson, Townsend, Zeleny (*Phil. Mag.*, 1898, [5], xlv., 120; see also *Amer. Chem. Journ.*, 1901, xxv., 340, for a résumé of this work), and others seem to indicate that the bearer of the negative electricity not only carries the high charge referred to above, but that it is very small, while the bearer of the positive electricity is very large. May it not be the atom itself which thus expands and contracts? This agrees with the verdict of the results of atomic compression given above. Change of atomic volume seems to be associated with electric stress. This assignment of electric expansibility to the atomic sphere of influence might explain other phenomena concerning the behaviour of electrified gases; for example, the increase of pressure which is observed when a gas is highly charged (De la Rue and Müller, *Phil. Trans.*, 1880, 86). Again, the great conductivity of a gas with adequate potential and quantity of electrical discharge seems to indicate that then the situation must resemble that in a metal, where the spheres of stress fill the whole volume occupied by the substance (Trowbridge and Richards, *Phil. Mag.*, 1897, [5], xliii., 349). The temperature must be so high under these circumstances that the gas is probably in a condition of thermal dissociation. Hence one is inclined to refer the great conductivity to the electrical susceptibility of evenly compressed or undistorted atoms. The fact that pure metals conduct electricity better than alloys or compounds seems to support this conclusion. The permeability of solids to cathode rays might be explained by supposing that the smallest particles of both solid and gas are much contracted by the negative charge.

It is with some diffidence that this paper attempts to reconcile the facts with any hypothesis, for hypotheses sometimes lead to dangerous delusions. If, however, one never forgets the essential difference between fact and hypothetical inference, a theory may afford useful suggestions for further research. The facts under discussion in the present paper seem to me to be adequately connected by none of the current conceptions concerning

atoms, hence it has seemed not wholly pointless to postulate a theory which might serve better. The essential elements of this theory must be evident from the trend of the hypothetical discussion above; they are not wholly new. Since changes of atomic volume seem to be so closely associated with the most intimate properties of substance, it seems necessary to assign more importance to the atomic “sphere of influence” or the “free space” around the atomic centres than is customary. Indeed, the properties of material seem to be as much concerned with the “atomic shell” as with the “atomic centre.” The two hypothetical conceptions are so closely related as to be inseparable.

Such a point of view leads to the conception of an atom as a compressible field of force possessing two attractive attributes, chemical affinity and gravitation, both of which may be concerned in chemical action. Mass may be supposed to be casually connected with gravitation. The fact that in many cases affinity diminishes with increasing atomic weight,* taken together with the Laws of Faraday and of Dulong and Petit, suggests that the two attractive forces in the atom may bear some sort of reciprocal or additive relationship to one another—that the product or sum of the two may afford a constant basis for the vibrations of heat and electricity. This relation is often hidden by electrical attraction, which plays so important a rôle in chemical action that it is sometimes hard to distinguish the intensity of chemical affinity proper. In such an atom one can imagine that either thermal or electrical vibration might cause distention. The phenomena of electricity suggest that electricity plays around the atomic surface, while heat seems to be concerned with a more fundamental or central agitation. Light-vibration, which seems also to be intimately concerned with atomic structure, would be assumed to be a surface effect like electrical vibration.

Such an atom would be compressible under the influence of its own affinities as well as under the influence of external pressure. Permanent atomic distortion would accompany chemical union, and the heat of the reaction would be the outcome of the resulting decrease of internal energy. Atomic volume and atomic compressibility might limit the possibility of distortion; hence would arise a possible explanation for quantivalence, stereochemistry, and crystal form. Many other properties of materials, too numerous to mention, seem to be explicable in a similar way.

It would be unreasonable to expect the hypothesis thus briefly described to correspond to all known facts. No hypothesis has ever been proposed which is wholly satisfactory; our knowledge is incommensurate with the possibilities involved. If, however, a given theory is found to explain some relationships better than other hypotheses, it may be of service in suggesting new experimental research. Such a service is of course the best one which a hypothesis can perform.

The idea discussed above has been already applied in plausible fashion to a wide range of chemical and physical phenomena. If future experimentation to be carried on here seems to warrant it, these applications may form the subject of another communication.

The object of the present paper may be summed up in a few words, as follows:—It is pointed out that changing atomic volume may be used as an approximate measure of the pressure which causes it, and therefore of the affinity which causes the pressure. Some of the difficulties in the way of exact interpretation are pointed out, and hints are given as to possible modes of overcoming the difficulties.

The chief outcome of the paper is the following postulate:—*The atomic volume is not constant, but a function of pressure and temperature, and probably of electric stress.*

* Van't Hoff, “Vorl. Th. Phys. Chem.,” III., 1900, 87. Compare also the relation of the energy-quotients of similar metals referred to in the present paper (p. 83).

In this connection it is pointed out that chemical affinity is possibly a reciprocal function of mass.

To explain these and many other facts, a modification of the atomic hypothesis is tentatively proposed which contends that we have no right to disregard the compressible environments around the centres of gravity and affinity.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING JULY 31ST, 1902.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolitan Water Act, 1871.

London, August 11th, 1902.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 216 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from July 1st to July 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 216 samples examined by us chemically during the month, all were found to be clear, bright, and well filtered.

The prolonged drought still continues; the actual rainfall at Oxford during July was 0.62 inch, the average fall is 2.49 inches; this leaves a deficit of 1.87 inches, which, added to the previous one of 3.30 inches, gives a total deficit of 5.17 inches, or 38.8 per cent on the thirty-five years' average.

Our bacteriological examinations of 562 samples have given the results recorded in the following table; we have also examined 40 other samples, from special standpipes, wells, &c., making a total of 602 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 27 samples) ..	292
New River, filtered (mean of 140 samples) ..	7
Thames, unfiltered (mean of 27 samples) ..	2172
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 304 samples)	14
Ditto ditto highest	212
Ditto ditto lowest	0
River Lea, unfiltered (mean of 27 samples) ..	250
River Lea, from the East London Company's clear-water wells (mean of 37 samples) ..	44

Of the 481 samples taken daily from the filter-wells of the Metropolitan Water Companies, and examined bacteriologically by us, forty samples, or 9.7 per cent, were sterile. Only three samples contained more than 150 microbes, while but twelve samples, or 2.4 per cent, contained more than 100 microbes per c.c. The mean number of microbes in the twelve excess samples was 149, against a corresponding mean of 206 in twenty-five samples during June.

The above results show that the water supplied to London during July was of exceptional purity, having regard to the average good quality of the London supply. It will be seen that the number of microbes is remarkably small, no less than 40 of the samples examined bacteriologically being sterile. But it must be remembered that the season is quite abnormal as regards rainfall, so that the Thames is delivering, practically, a considerable proportion of spring water.

We are, Sir,
Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

NOTICES OF BOOKS.

Essays on Historical Chemistry. By T. E. THORPE, C.B., LL.D., F.R.S., Principal of the Government Laboratory, London. London: Macmillan and Co., Ltd. New York: The Macmillan Company. 1902. Pp. 582.

The present volume consists principally of lectures and addresses given at different times by the author during the last twenty-five years; these essays are now put together with the object of showing how the labours of some of the greatest masters of chemical science have contributed to its development. The book is mainly biographical, and includes short sketches of the lives of such men as Priestley, Cavendish, Lavoisier, Faraday, Dumas, Victor Meyer, Mendeleeff, and others.

"The Rise and Development of Synthetical Chemistry," which forms the subject of No. XVI. of this series, is of considerable interest; it first saw light as the Presidential Address to the Sutton Coldfield Institute in the year 1892, and traces the advance of synthetical chemistry from the first inception of the atomic hypothesis down to the triumphs of recent years, as illustrated by the artificial formation of indigo and that of the sugars, *dextrose* and *levulose*. The advance in every section of chemistry during the nineteenth century, and especially during the latter half of it, has been literally by leaps and bounds, and no branch has been more fruitful in results or possibilities than that of organic synthesis.

No. XVII., on "The Progress of Chemistry in Great Britain and Ireland during the Nineteenth Century," covers the ground from the beginning of the last century to the foundation of the Chemical Society in 1841; and No. XVIII., on "The Development of the Chemical Arts during the Reign of Queen Victoria," brings us up to comparatively recent times.

The book is well worth looking through, and contains a large amount of interesting matter.

Higher Mathematics for Students of Physics and Chemistry. By J. W. MELLOR, D.Sc. London: Longmans, Green, and Co. 1902.

This book is intended for those who require a practical knowledge of higher mathematics to enable them to deal with, and interpret from a mathematical point of view, the results of physical and chemical experiments. The reader is supposed to possess only a very elementary knowledge of algebra and trigonometry, and even this minimum is recapitulated for the benefit of those who have forgotten their school-work. The book is divided into three parts. Part I. (Elementary) deals with the Differential Calculus, Analytical Geometry, and the Integral Calculus. As far as possible, all examples are based upon figures obtained in actual experiments or well-known formulae, and thus no opportunity is lost of incidentally adding to chemical knowledge. As is to be expected, the discussion of the integral calculus leads to most interesting pages on such branches of the subject as

chemical equilibrium, fractional precipitation, &c. The second part is occupied with advanced mathematics, and opens with a chapter on hyperbolic functions, and thence passes to the consideration of differential equations; this part will undoubtedly be found most useful, for the subject is treated quite fully enough for the purpose in view, and the student is saved from the necessity of making generally fruitless attempts to grapple with such difficult, though admirable, text-books as Forsyth's on the subject. The third part contains short expositions of the theories of equations, determinants, probability, and errors; this last forming one of the best and most interesting chapters in the book. A collection of useful results and formulæ is appended. The least mathematical of science students cannot help finding this volume attractive and useful; it may safely be said to have thoroughly fulfilled its aim, and will no doubt soon be regarded as indispensable to the student and teacher alike. The style in which it is written is very far removed from that generally associated with a mathematical text-book, and the freedom and graphic directness of some of the writing will do much towards investing the subject with a new interest.

Berichte über Land- und Forstwirtschaft in Deutsch-Ostafrika ("Reports on Agriculture and Forestry in German East Africa"). Band I. Hefte 1 and 2. Herausgegeben vom Kaiserlichen Gouvernement von Deutsch-Ostafrika. Heidelberg: Carl Winter's Universitätsbuchhandlung. 1902.

THESE Government reports of the Colonial department of the Foreign Office deal with questions relating to agriculture, and contain also discussions and articles on such subjects as the diseases of animals and plants, meteorological conditions, geography, and geology. The first number consists of extracts from the annual report for the year July 1, 1900, to June 30, 1901, of the district and military stations in various regions of German East Africa. These are chiefly concerned with agricultural activity, the export and import trades, and the results of the experimental cultivation of various foreign plants and trees. The second number contains articles mainly of biological interest, with the exception of the reports of chemical researches on some soils in the neighbourhood of Tanga in Usambara. Numerous analyses of the soil at various depths were made, and the results are tabulated here. With a few exceptions these results point to the fact that the soil of this region of German East Africa is singularly good.

Beiträge zur Chemischen Physiologie und Pathologie. ("Contributions to Chemical Physiology and Pathology"). Edited by FRANZ HOFMEISTER. Band II. 7-9 Heft. Braunschweig: Friedrich Vieweg und Sohn. 1902.

THIS number contains papers of much chemical interest. Amongst them may be specially mentioned Dr. B. Slowtsoff's report of his investigations of the action of the liver cells on copper, a continuation of his previously described researches on similar actions on arsenic and mercury. He establishes the fact that, like arsenic, copper, when administered in the form of sulphate, forms a compound with the nuclein of the liver; this compound, however, is attacked by 0.3 per cent hydrochloric acid, and destroyed by pepsin hydrochloric acid, and thus is not so stable as the arsenic compound. A paper by F. Samuely on melanins derived from albumen represents an immense amount of apparently most thorough and careful experimental work. The chemical behaviour of the artificial melanins has been subjected to an ably conducted inquiry, and the paper contains much useful information. Another article by R. Schröder on the protein substances of yeast is full of interest, though some of the author's results call for further investigation on the same lines.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxv., No. 3, July 21, 1902.

Action of Hydrochloric Acid on the Sulphates of Aluminium, Chromium, and Iron Sesquioxides.—A. Recoura.—The author's experiments on the behaviour of solutions of the salts of sesquioxides of aluminium, chromium, and iron, when they are reacted upon by an acid different from that of the salt. He finds that the sulphate of chromium sesquioxide, for example, under the action of heat, dissolves a portion of the sulphuric acid set free. The result of this decomposition in presence of another acid weaker than sulphuric acid, such as hydrochloric acid, when used in great excess, has the power of fixing one or more molecules of hydrochloric acid, giving rise to a polyacid salt in which the hydroxyls of the base are saturated, some by the sulphuric, others by the hydrochloric acid. By using this method the author obtained the two polyacid salts, $\text{AlSO}_4\text{Cl}_6\text{H}_2\text{O}$ and $\text{CrSO}_4\text{Cl}_6\text{H}_2\text{O}$, whilst with the sulphate of iron he transformed ferric sulphate, Fe_2SO_4 , into an acid sulphate, $\text{Fe}_2\text{SO}_4\text{SO}_4\text{H}_2\text{SO}_4$.

Precipitation of Cupric Chloride and Bromide with Sulphuric Acid.—Georges Viard.—An excess of concentrated sulphuric acid gives a yellowish-brown precipitate of the anhydrous chloride when added to a solution of cupric chloride, and a black precipitate of the anhydrous bromide when added to cupric bromide. The same precipitates are produced by adding an excess of sulphuric acid to any cupric salt in presence of an alkaline chloride or bromide. This property gives a convenient distinction between chlorides and bromides, the best application being to prepare beforehand a mixture of 1 volume of copper sulphate with 10 volumes of H_2SO_4 . By adding to this reagent a few drops of the salt to be examined a yellow precipitate is formed if a chloride is present, a black precipitate if it is a bromide. Very dilute solutions may thus easily be recognised.

Cerium Silicide.—M. Sterba.—The author obtains a cerium silicide of formula CeSi_2 , different from M. Ulrik's silicide. Its relative stability allows of its preparation in Moissan's electric furnace. Its properties are different from those of calcium silicide. It seems to approach the silicides of the heavy metals in properties.

Action of Alcohols on the Sodium Derivatives of other Alcohols.—Marcel Guéret.—As a result of a previous research the author predicts the condensation occurring between certain alcohols and the sodium derivatives of other alcohols, e.g., between ethylic or propylic alcohols, and the sodium derivative of ceanthylic alcohol. The reaction can be expressed by the equation:—

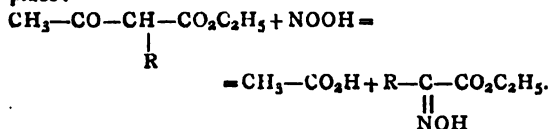


Investigation on the Simultaneous Distillation of two Non-miscible Substances.—Eug. Charabot and J. Rocherolles.—The two laws which the authors establish during the present research are contained in the following statement:—The proportion between the weight of a body non-miscible with water and the weight of water which simultaneously distills, varies in such a manner as to approach unity when the temperature increases without attaining the critical temperature of one of the two substances.

A New Di-iodated Phenol.—P. Brenans.—The author prepares a new isomer of di-iodated phenol, $\text{OH}-\text{C}_6\text{H}_3\text{I}_2$ 1.3.6, from orthonitraniline. By mixing solutions of iodine chloride and orthonitraniline in acetic acid he prepares mono-iodated orthonitraniline,

$C_6H_5(NH_2)(NO_2)(I)_2$ 2.4. The diazoic derivative of this last body can be decomposed by means of potassium iodide, and gives a di-iodated nitrobenzene, $C_6H_5(NO_2)(I)_2$ 1.3.6. The corresponding base, di-iodated aniline, $C_6H_5(NH_2)(I)_2$ 1.3.6, gives by diazotization and decomposition of the diazoic radical in presence of water, di-iodophenol, $OH-C_6H_3I_2$ 1.3.6.

Action of Nitrous Acid in Acid Solution on the β -Ketonic- α -substituted Ethers. Synthesis of the Homologues of Pyruvic Acid.—L. Bouveault and R. Locquin.—The authors cause nitrous acid, not in alkaline solution, as previous experimenters have done, but in acid solution, to react on the α -substituted acetylacetic ethers, and they have established the fact that under these conditions it is always the following reaction which takes place:—



The reactions are perfectly definite. Nitrosation in acid solution of the β -ketonic- α -substituted ethers by any radical whatever, primary or secondary, yields exclusively, by separation of the acid radical, the oximes of substituted glyoxylic ethers. From this a very convenient method of preparing a great number of ethers of the homologues superior to the pyruvates can be deduced.

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Candidates who have complied with the Regulations, and desire to present themselves for this Examination, are required to communicate with the Registrar before Tuesday, September 23rd next, when the List of Candidates for this Examination will be closed.

By Order of the Council,
RICHARD B. PILCHER, Registrar.
30, Bloomsbury Square, London, W.C., August 11, 1902.

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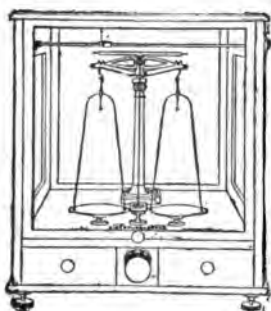
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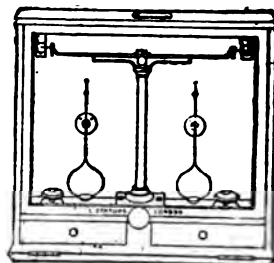


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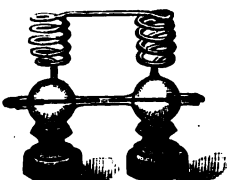
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THE CHEMICAL NEWS.

VOL. LXXXVI., No. 2231.

THE RADIO-ACTIVITY OF THORIUM COMPOUNDS.*

II. THE CAUSE AND NATURE OF RADIO-ACTIVITY.

By E. RUTHERFORD, M.A., D.Sc.,
Macdonald Professor of Physics, McGill University, Montreal,
and
FREDERICK SODDY, B.A. (Oxon.),
Demonstrator in Chemistry, McGill University, Montreal.

(Continued from vol. lxxxv., p. 308).

AT the close of the first paper on this subject (*Trans. Chem. Soc.*, 1902, p. 321; *CHEMICAL NEWS*, vol. lxxxv., p. 271 *et seq.*) it was shown that a constituent responsible for part of the radio-activity of thorium could be separated from its compounds by chemical means. Two methods were given. In one a thorium solution was precipitated by ammonia, and the thorium hydroxide precipitate showed only about one-third of the normal activity, while the filtrate on evaporation and removal of the ammonium salts by ignition left a very active residue—in some cases over a thousand times as active as an equal weight of thorium. In the other method, thorium oxide was washed repeatedly with large quantities of water. The washings on evaporation deposited very active residues, while the radio-activity of the thorium was appreciably diminished by the process.

In both of these methods the active residues are extremely small, and the view was put forward that even the most active specimens consisted largely of accidental impurities, ThX—the active constituent of thorium—being only present in minute amount. By the kindness of Dr. Knöfler, of Berlin, who in the friendliest manner placed at our disposal a large specimen of his purest thorium nitrate, we were at once able to confirm this opinion. This specimen, which had been purified by a great many processes, did not contain any of the impurity precipitable by sodium phosphate after the removal of the thorium with ammonia, which was present in the commercial nitrate before used. The radio-activity and emanating power of Dr. Knöfler's specimen were, however, at least as great as any other in our possession, and the residues obtained from the filtrate after precipitating with ammonia were no less active.

1. Scope of the Present Paper.

In the present communication the results of a further detailed investigation of the radio-activity and emanating power of thorium compounds are given, and these have led to a theoretical interpretation of the processes involved which give rise to the phenomenon of natural radio-activity.

The Influence of Time on the Activity of Thorium and ThX.—The preparations employed in our previous experiments were allowed to stand over during the Christmas vacation. On examining them about three weeks later it was found that the thorium hydroxide which had originally possessed only about 36 per cent of its normal activity had almost completely recovered the usual value. The active residues, on the other hand, prepared by both methods, had almost completely lost their original activity. The chemical separation effected was thus not permanent in character. At this time, M. Becquerel's paper (*Comptes Rendus*, cxxxiii., p. 977, December 9, 1901) came to hand, in which he shows that the same phenomena of recovery and decay are presented by uranium after it has been partially separated from its active constituent by chemical treatment.

* Communicated by the Authors.

A long series of observations was at once started to determine:—

1. The rate of recovery of the activity of thorium rendered less active by removal of ThX;
2. The rate of decay of the activity of the separated ThX;

in order to see how the two processes were connected. The results led to the view that may at once be stated. The radio-activity of thorium at any time is the resultant of two opposing processes.

1. The production of fresh radio-active material at a constant rate by the thorium compound;
2. The decay of the radiating power of the active material with time.

The normal or constant radio-activity possessed by thorium is an equilibrium value, where the rate of increase of radio-activity due to the production of fresh active material is balanced by the rate of decay of radio-activity of that already formed. It is the purpose of the present paper to substantiate and develop this hypothesis.

2. The Rates of Recovery and Decay of Thorium Radio-activity.

A quantity of the pure thorium nitrate was separated from ThX in the manner described by several precipitations with ammonia. The radio-activity of the hydroxide so obtained was tested at regular intervals to determine the rate of recovery of its activity. For this purpose the original specimen of 0.5 gm. was left undisturbed throughout the whole series of measurements on the plate over which it had been sifted, and was compared always with 0.5 gm. of ordinary "de-emanated" thorium hydroxide spread similarly on a second plate, and also left undisturbed. The emanation from the hydroxide was prevented from interfering with the results by a special arrangement for drawing a current of air over it during the measurements.

The rate of rise of emanating power of the same preparation was determined at the same time (see Section 7). The methods and apparatus employed have already been described in the previous paper.

The active filtrate from the preparation was concentrated and made up to 100 c.c. volume. One quarter was evaporated to dryness, and the ammonium nitrate expelled by ignition in a platinum dish, and the radio-activity of the residue tested at the same intervals as the hydroxide to determine the rate of decay of its activity. The comparison in this case was a standard sample of uranium oxide kept undisturbed on a metal plate, which repeated work has shown to be a perfectly constant source of radiation. The remainder of the filtrate was used for other experiments (Sections 5 and 7).

The following table gives an example of one of a numerous series of observations made with different preparations at different times. The maximum value attained by the hydroxide and the original value of the ThX are taken as 100:—

Time in days.	Activity of hydroxide.	Activity of ThX.
0	44	100
1	37	117
2	48	100
3	54	88
4	62	72
5	68	—
6	71	53
8	78	—
9	—	29.5
10	83	25.2
13	—	15.2
15	—	11.5
17	96.5	—
21	99	—
28	100	—

Fig. 1 shows the curves obtained by plotting the radio-activities as ordinates, and the time in days as abscissæ.

Curve 2 illustrates the rate of recovery of the activity of thorium. Curve 1 the rate of decay of activity of ThX. It will be seen that neither of the curves is regular for the first two days. The activity of the hydroxide at first actually diminished, and was at the same value after two days as when first prepared. The activity of the ThX, on the other hand, at first increases, and does not begin to fall below its original value till after the lapse of two days (compare Section 8). These results cannot be ascribed to errors of measurement, for they have been regularly observed whenever similar preparations have been tested. The activity of the residue obtained from thorium oxide by the second method of washing decayed very similarly to that of ThX as shown by the above curve.

If for present purposes the initial periods of the curve are disregarded and the later portions only considered, it will be seen at once that the time taken for the hydroxide to recover one-half of its lost activity is about equal to the time taken by the ThX to lose half its activity, viz., in each case about four days, and, speaking generally, the percentage proportion of the lost activity regained by the hydroxide over any given interval is approximately equal to the percentage proportion of the activity lost by the ThX during the same interval. If the recovery curve is produced backwards in the normal direction to cut the vertical axis, it will be seen to do so at a minimum of about 25 per cent, and the above result holds even more accurately if the recovery is assumed to start from this constant minimum, as indeed it has been shown to do under suitable conditions (Section 8, Fig. 4). This is brought out by Fig. 2, which represents the recovery curve of thorium in which the percentage amounts of activity recovered, reckoned from this 25 per cent minimum, are plotted as ordinates. In the same figure the decay curve after the second day is shown on the same scale.

The activity of ThX decreases very approximately in a geometrical progression with the time, i.e., if I_0 represent the initial activity and I_t the activity after time t ,

$$\frac{I_t}{I_0} = e^{-\lambda t} \quad \dots \dots \dots (1),$$

where λ is a constant and e the base of natural logarithms.

The experimental curve obtained with the hydroxide for the rate of rise of its activity from a minimum to a maximum value will therefore be approximately expressed by the equation—

$$\frac{I_t}{I_0} = 1 - e^{-\lambda t} \quad \dots \dots \dots (2),$$

where I_0 represents the amount of activity recovered when the maximum is reached, and I_t the activity recovered after time t , λ being the same constant as before.

Now, this last equation has been theoretically developed in other places (compare Rutherford, *Phil. Mag.*, 1900, pp. 10 and 181) to express the rise of activity to a constant maximum of a system consisting of radiating particles in which:—

1. The rate of supply of fresh radiating particles is constant;
2. The activity of each particle dies down geometrically with the time according to equation (1).

It therefore follows that if the initial irregularities of the curves are disregarded and the residual activity of thorium is assumed to possess a constant value, the experimental curve obtained for the recovery of activity will be explained if two processes are supposed to be taking place:—

1. That the active constituent ThX is being produced at a constant rate;
2. That the activity of the ThX decays geometrically with time.

Without at first going into the difficult questions connected with the initial irregularities and the residual activity, the main result that follows from the curves given can be put to experimental test very simply. The primary conception is that the major part of the radio-

activity of thorium is not due to the thorium at all, but to the presence of a non-thorium substance in minute amount which is being continuously produced.

3. Chemical Properties of ThX.

The fact that thorium on precipitation from its solutions by ammonia leaves the major part of its activity in the filtrate, does not of itself prove that a material constituent responsible for this activity has been chemically separated. It is possible that the matter constituting the non-thorium part of the solution is rendered temporarily radio-active by its association with thorium, and this property is retained through the processes of precipitation, evaporation, and ignition, and manifests itself finally on the residue remaining.

This view, however, can be shown to be quite untenable, for upon it any precipitate capable of removing thorium completely from its solution should yield active residues similar to those obtained from ammonia. Quite the reverse, however, holds.

When thorium nitrate is precipitated by sodium or ammonium carbonate, the residue from the filtrate by evaporation and ignition is free from activity, and the thorium carbonate obtained possesses the normal value for its activity.

The same holds true when oxalic acid is used as the precipitant. This reagent, even in strongly acid solution, precipitates almost all of the thorium. When the filtrate is rendered alkaline by ammonia, filtered, evaporated, and ignited, the residue obtained is inactive.

In the case where sodium phosphate is used as the precipitant in ordinary acid solution, the part that comes down is more or less free from ThX. On making the solution alkaline with ammonia, the remainder of the thorium is precipitated as phosphate, and carries with it the whole of the active constituent, so that the residue from the filtrate is again inactive.

In fact, ammonia is the only reagent of those tried capable of separating ThX from thorium.

The result of Sir William Crookes with uranium, which we have confirmed working with the electrical method, may be here mentioned. UrX is completely precipitated by ammonia, together with the uranium, and the residue obtained by the evaporation of the filtrate is quite inactive.

There can thus be no question that both ThX and UrX are distinct types of matter with definite chemical properties. Any hypothesis that attempts to account for the recovery of activity of thorium and uranium with time must of necessity start from this primary conception.

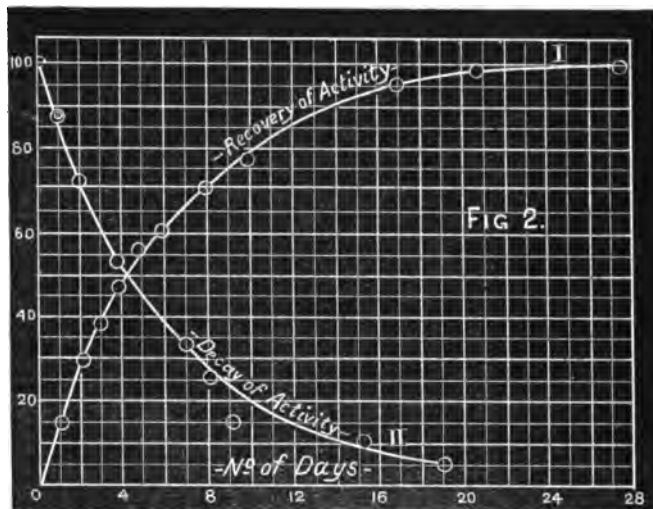
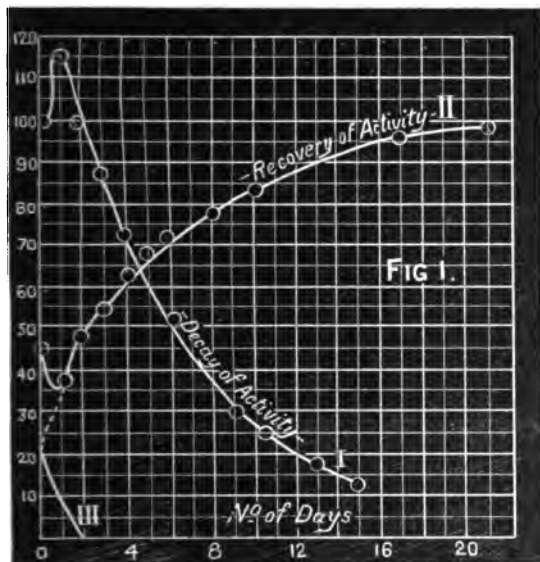
4. The Continuous Production of ThX.

If the recovery of the activity of thorium with time is due to the production of ThX, it should be possible to obtain experimental evidence of the process. The first point to be ascertained is how far the removal of ThX by the method given reduces the total radio-activity of thorium. A preliminary trial showed that the most favourable conditions for the separation was by precipitating in hot dilute solutions by dilute ammonia. A quantity of 5 grms. of thorium nitrate, as obtained from the maker, was so precipitated by ammonia, the precipitate being re-dissolved in nitric acid, and re-precipitated under the same conditions successively *without lapse of time*. The removal of ThX was followed by measuring the activity of the residues obtained from the successive filtrates. The activity of the ThX from the first filtrate was equivalent to 4.23 grms. of thorium, from the second to 0.33 grm., and from the third to 0.07 grm. It will be seen that by two precipitations the whole of the ThX is removed. The radio-activity of the separated hydroxide was 48 per cent of that of the standard de-emanated sample of thorium.

Rate of Production of ThX.—A quantity of thorium nitrate solution, that had been freed from ThX about a month before, was again subjected to the same process.

The activity of the residue from the filtrate in an experiment in which 10 grms. of this nitrate had been employed was equivalent to 8.3 grms. of thorium oxide. This experiment was performed on the same day as the one recorded above, in which 5 grms. of new nitrate had been employed, and it will be seen that there is no difference in the activity of the filtrates in the two cases. In one month

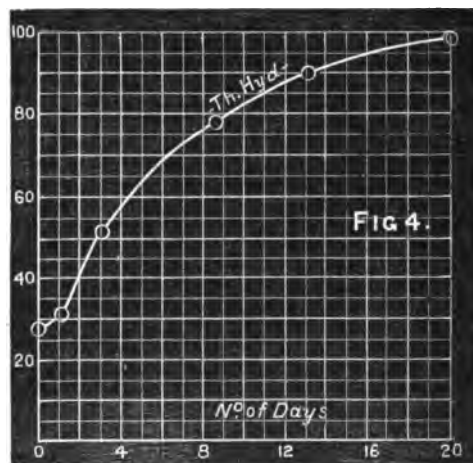
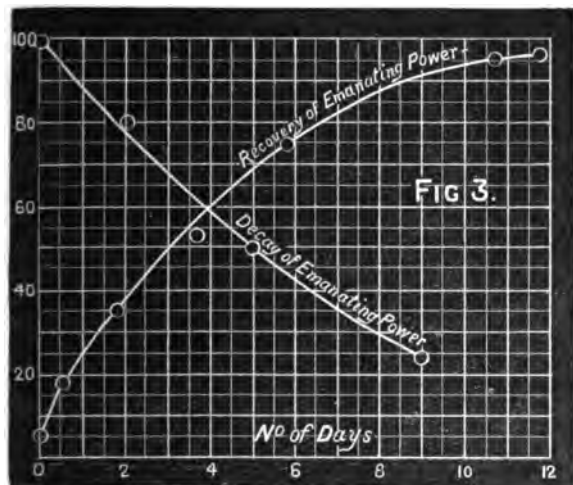
effect, the course of production of ThX can be followed after extremely short intervals. Working with 10 grms. of thorium nitrate, the amount produced in the minimum time taken to carry out the successive precipitations is as much as can be conveniently measured. If any interval is allowed to elapse the effect is beyond the range of the instrument, unless the sensitiveness is



the activity of the ThX in a thorium compound again possesses its maximum value.

If a period of two hours is allowed to elapse between the successive precipitations, the activity of the ThX formed during that time corresponds to about one-sixth of the maximum activity of the total thorium employed. In three hours the activity of the amount produced is about one-thirtieth. The rate of production of ThX worked out

reduced to a fraction of its ordinary value by the introduction of capacities into the system. Capacities of 0.01 and 0.02 microfarad, which reduce the sensitiveness to less than one-hundredth and one two-hundredth of the normal, were frequently employed in dealing with these active residues. For ordinary work with 0.25 to 0.5 gm. of thorium compound 0.001 microfarad was necessary. Most of the measurements in the course of the present paper



from these figures well agrees with the form of the curve obtained for the recovery of activity of thorium, if the latter is taken to express the continuous production of ThX at a constant rate and diminution of the activity of the product in geometrical progression with the time.

By using the Dolezalek electrometer, on which the radio-activity of 1 m.grm. of thorium produces a measurable

were made with this instrument, and a range from that represented by 1 m.grm. of thorium to any desired maximum could readily be obtained. Of course the greatest care is necessary in working with so sensitive an instrument to prevent electrostatic disturbances of every kind.

The process of the production of ThX is continuous, and no alteration was observed in the amount produced in a

given time after repeated separations. In an experiment carried out for another purpose (Section 8) after twenty-three successive precipitations extending over nine days, the amount formed during the last interval was, as far as could be judged, no less than what occurred at the beginning of the process.

The phenomenon of radio-activity, by means of the electrometer as its measuring instrument, thus enables us to detect and measure changes occurring in matter after a few minutes interval, which have never yet been detected by the balance or suspected of taking place.

5. Influence of Conditions on the Changes occurring in Thorium.

It has been shown that in thorium compounds the decay of radio-activity with time is balanced by a continuous production of fresh active material. The change which produces this material must be chemical in nature, for the products of the action are different in chemical properties from the thorium from which they are produced. The first step in the study of the nature of this change is to examine the effect of conditions upon its rate.

Effect of Conditions on the rate of Decay.—Since the activity of the products affords the means of measuring the amount of change, the influence of conditions on the rate of decay must first be found. It was observed that like all other types of temporary radio-activity the rate of decay is unaltered by any known agency. It is unaffected by ignition and chemical treatment, and the material responsible for it can be dissolved in acids, and re-obtained by the evaporation of the solution without affecting the activity. The following experiment shows that the activity decays at the same rate in solutions as in the solid state:—The remainder of the solution that had been used to determine the decay curve of ThX (Fig. 1) was allowed to stand, and at the end of twelve days a second quarter was evaporated to dryness and ignited, and its activity compared with that of the first, which had been left since evaporation upon its original platinum dish. The activities of the two specimens so compared with each other was indistinguishable, showing that in spite of the very different conditions the two fractions had decayed at equal rates. After nineteen days a third quarter was evaporated, and the activity, now very small, was indistinguishable from that of the fraction first evaporated. Re-solution of the residue after the activity had decayed does not at all regenerate it. The activity of ThX thus decays at a rate independent of the chemical and physical condition of the molecule.

Therefore the rate of recovery of activity under different conditions in thorium compounds affords a direct measure of the rate of production of ThX under these conditions. The following experiments were performed:—

One part of thorium hydroxide newly separated from ThX was sealed up in a vacuum obtained by a good Töpler pump, and the other part exposed to air. On comparing the samples twelve days later, no difference could be detected between them either in their radio-activity or emanating power.

In the next experiment a quantity of hydroxide freed from ThX was divided into two equal parts; one was exposed for twenty hours to the heat of a Bunsen burner in a platinum crucible, and then compared with the other. No difference in the activities was observed. In a second experiment one-half was ignited for twenty minutes on the blast, and then compared with the other with the same result. The difference of temperature and the conversion of thorium hydroxide into oxide thus exercised no influence on the activity.

Some experiments that were designed to test in as drastic a manner as possible the effect of the chemical condition of the molecule on the rate of production of ThX brought to light small differences, but these are almost certainly to be accounted for in another way. It will be shown later (Section 8) that about 21 per cent of the normal radio-activity of thorium oxide under ordinary con-

ditions consists of a secondary activity excited on the mass of the material. This portion is of course a variable, and since it is divided among the total amount of matter present, the conditions of aggregation, &c., will affect the value of this part. This effect of excited radio-activity in thorium makes a certain answer to the question difficult, and on this account the conclusion that the rate of production of ThX is independent of the molecular conditions is not final. The following experiment, however, makes it extremely probable:—

A quantity of thorium nitrate as obtained from the maker was converted into oxide in a platinum crucible by treatment with sulphuric acid and ignition to a white heat. The de-emanated oxide so obtained was spread on a plate, and any change in radio-activity with time, which under these circumstances could certainly be detected, was looked for during the first week from preparation. None whatever was observed, whereas if the rate of production of ThX in thorium nitrate is different from that in the oxide, the equilibrium point at which the decay and increase of activity balance each other will be altered in consequence. There should have therefore occurred a logarithmic rise or fall from the old to the new value. As, however, the radio-activity remained constant, it appears very probable that the changes involved are independent of the molecular condition. It will be seen that the assumption is here made that the proportion of excited radio-activity in the two compounds is the same, and for this reason compounds were chosen which possess but low emanating power. (Compare Section 7, last paragraph).

Uranium is a far simpler example of a radio-active element than thorium, as the phenomena of excited radio-activity and emanating power are here absent. The separation of UrX and the recovery of the activity of the uranium with time appear, however, analogous to these processes in thorium, and the rate of recovery and decay of uranium activity are at present under investigation. It is proposed to test the influence of conditions on the rate of change more thoroughly in the case of uranium, as here secondary changes do not interfere.

6. The Cause and Nature of Radio-activity.

The foregoing conclusions enable a great generalisation to be made in the subject of radio-activity. Energy considerations require that the intensity of radiation from any source should die down with time unless there is a constant supply of energy to replace that dissipated. This has been found to hold true in the case of all known types of radio-activity with the exception of the "naturally" radio-active elements—to take the best established cases, thorium, uranium, and radium. In their first paper on the present subject, the authors showed that the radio-activity of the emanation produced by thorium compounds decayed geometrically with the time under all conditions, and was not affected by the most drastic chemical and physical treatment. The same has been shown by one of us (*Phil. Mag.*, 1900, p. 161) to hold for the excited radio-activity produced by the thorium emanation. This decays at the same rate whether on the wire on which it is originally deposited, or in solution of hydrochloric or nitric acid. The excited radio-activity produced by the radium emanation appears analogous. All these examples satisfy energy considerations. In the case of the three naturally occurring radio-active elements, however, it is obvious that there must be a continuous replacement of the dissipated energy, and no satisfactory explanation has yet been put forward.

The nature of the process becomes clear in the light of the foregoing results. The material constituent responsible for the radio-activity, when it is separated from the thorium which produces it, then behaves in the same way as the other types of radio-activity cited. Its activity decays geometrically with the time, and the rate of decay is independent of the molecular conditions. The normal radio-activity is, however, maintained at a constant value by a chemical change which produces fresh radio-active

material at a rate also independent of the conditions. The energy required to maintain the radiations will be accounted for if we suppose that the energy of the system after the change has occurred is less than it was before.

The work of Crookes and Becquerel on the separation of Ux and the recovery of the activity of the uranium with time, makes it appear extremely probable that the same explanation holds true for this element. The work of M. and Mme. Curie, the discoverers of radium, goes to show that this body easily suffers a temporary decrease of its activity by chemical treatment, the normal value being regained after the lapse of time, and this can be well interpreted on the new view. All known types of radio-activity can thus be brought under the same category.

(To be continued.)

ON THE FERROCYANIDES OF CADMIUM.*

By EDMUND H. MILLER.

It has already been shown (Miller and Fisher, *Yourn. Amer. Chem. Soc.*, September, 1900) that the results obtained in titrating cadmium by potassium ferrocyanide in a neutral or slightly acid solution do not agree with any of the formulas given for the precipitate, while the values obtained in an ammoniacal solution agree very closely with the formula $K_2CdFe(CN)_6$.

The present work was undertaken to study the variation in composition under different conditions, and, if possible, to assign definite formulas to the precipitates so formed. To cover the ground as completely as possible, the following series of precipitates were made:—

- A.—Neutral solution, cadmium in excess.
- B.—Neutral solution, ferrocyanide in excess.
- C.—Acid with hydrochloric, 10 c.c. (sp. gr. 1.20) per litre, cadmium in excess.
- D.—Acid with hydrochloric, 10 c.c. (sp. gr. 1.20) per litre, ferrocyanide in excess.
- E.—Acid with acetic, 10 c.c. 50 per cent acetic acid per litre, cadmium in excess.
- F.—Acid with acetic, 10 c.c. 50 per cent acetic acid per litre, ferrocyanide in excess.
- G.—Alkaline with ammonia,† cadmium in excess.
- H.—Alkaline with ammonia,† ferrocyanide in excess.

The precipitates were all formed in large beakers, in the cold, and allowed to stand, then washed with water, water containing hydrochloric acid, water containing acetic acid, water and ammonia, respectively, so that the conditions of precipitation were maintained, then washed with water till free from excess of cadmium or of ferrocyanide. The work was begun in June, 1900, and the precipitates analysed a year later.

In general, whenever the cadmium was in excess the precipitates settled well, and when ferrocyanide was in excess, very badly. The salts used for this series of precipitations were cadmium nitrate and potassium ferrocyanide.

The method of analysis was in all cases the same, and the object was to obtain the ratio between iron and potassium and cadmium. Some attempts were made at first to dry the precipitates to constant weight, but as decomposition resulted no attempt was made to weigh the portions taken for analysis, but all the determinations were expressed as ratios.

The precipitates were repeatedly evaporated in casseroles with nitric and sulphuric acids till decomposition was complete, the residue was dissolved in water, the excess of sulphuric acid neutralised with ammonia, and the cadmium precipitated as sulphide in a very weak

hydrochloric acid solution, dissolved and determined as phosphate (Miller and Page, *School of Mines Quarterly*, July, 1901, and *Zeit. Anorg. Chem.*, 1901, xxviii., 233). In the filtrate the iron was, after oxidation, precipitated as hydroxide and weighed as ferric oxide. The filtrate from the iron was evaporated, the ammonium salts expelled, and the potassium weighed as sulphate. In the determination of cadmium as phosphate, weighing as pyrophosphate, and as cadmium ammonium phosphate, $(CdNH_4PO_4 \cdot H_2O)$, were tried, and the weighing as cadmium ammonium phosphate adopted as more convenient.

The following is a brief description of the precipitates formed and the results obtained from their analysis expressed as an atomic ratio* :—

A (neutral solution, cadmium in excess).—Yellowish-white, settled well, was washed (nine times) till free from cadmium; when dry, was yellowish-white, apparently slightly oxidised to ferricyanide. Gave, on analysis, $Fe:K:Cd::1:1.14:1.42$ or $7:8:10$, corresponding to $K_8Cd_{10}[Fe(CN)_6]_7$.

B (neutral, ferrocyanide in excess).—White, settled partially after five months, was washed at intervals for a year. It was the most tedious of all, but did not give evidence of decomposition. The ratio was $Fe:K:Cd::1:1.80:1.08$.

C (hydrochloric, cadmium in excess).—Dull orange-yellow, settled very well; on washing with water containing 10 c.c. concentrated hydrochloric acid per litre, the supernatant liquid became greenish with evident decomposition of the precipitate. A second lot was made under the same conditions; the precipitate, at first white, became yellow within a few seconds. The yellow precipitate is also formed when cadmium is dissolved in hydrochloric and a little nitric acid, then neutralised, 2 c.c. of hydrochloric acid added, and then titrated by potassium ferrocyanide. It is not due to the presence of any nitroprusside, and is undoubtedly due to the oxidation of part of the cadmium ferrocyanide to ferricyanide, which is brownish-yellow, by the nitrate present in both cases. This precipitate was not analysed.

D (hydrochloric, ferrocyanide in excess).—The precipitate, at first yellowish, became white when an excess of potassium ferrocyanide was present; it settled quite well at first, but badly after washing, like the others when an excess of ferrocyanide is present, and showed a greenish-yellow colour after washing, evidently due to a slight decomposition. The results were $Fe:K:Cd::1:1.933:0.991$, corresponding to $K_2CdFe(CN)_6$.

E (acetic, cadmium in excess).—The precipitate was nearly white, settled well, and on drying was white. The ratio was $Fe:K:Cd::1:1.21:1.392$, corresponding quite closely to $K_6Cd_7[Fe(CN)_6]_5$.

F (acetic, cadmium in excess).—Creamy white precipitate which settled badly, and on drying was slightly greenish on the surface. The iron cadmium ratio was $1:0.95$, corresponding approximately to $K_2CdFe(CN)_6$.

G (ammonia, cadmium in excess).—A pure white precipitate which gave considerable trouble in testing for an excess of cadmium, as owing to the excess of cadmium dissolved in ammonia, a point was reached where either $FeCN_6'''$ or Cd'' ions gave a precipitate. The precipitate on washing became flocculent, and settled immediately; it was washed six times with water containing ammonia, and then with water until no test for cadmium was obtained. The analytical results gave the ratio $Fe:K:Cd::1:0.333$, a result impossible for a ferrocyanide. After re-washing with strong ammonia to dissolve out the excess of cadmium, which was undoubtedly present as either hydroxide or carbonate, and which gave the flocculent character to the precipitate, the ratio be-

* Contribution from the Havemeyer Laboratories, Columbia University. From the *Journal of the American Chemical Society*, xiv., No. 3.

† The cadmium hydroxide was dissolved in an excess of ammonia before the potassium ferrocyanide was added.

* These values are obtained by dividing the calculated weights of iron, potassium, and cadmium, by their atomic weights, and then multiplying by $\frac{1}{wt. iron}$.

came Fe: Cd :: 1:1.94, agreeing approximately with the normal ferrocyanide, $\text{Cd}_2\text{Fe}(\text{CN})_6$.

H (ammonia, ferrocyanide in excess).—Pure white precipitate which settles well at first, but poorly after washing.* In this case only, the iron cadmium ratio was determined; the figures obtained were 1:0.97, corresponding to $\text{K}_2\text{CdFe}(\text{CN})_6$.

On account of the oxidation and decomposition of some of the precipitates just described, a second set of eight was made under exactly the same conditions, using cadmium chloride instead of nitrate. These were made in December, 1900, and are marked A', B', &c., so as to be readily compared with the preceding series.

A' (neutral, cadmium in excess).—White precipitate, which settled well, and was completely washed in a month. On analysis, the ratio found was Fe:K: Cd :: 1:1.14: 1.448 or 7:8:10, agreeing with the results obtained in A and the formula $\text{K}_8\text{Cd}_{10}[\text{Fe}(\text{CN})_6]_7$.

B' (neutral, ferrocyanide in excess).—A slightly yellowish precipitate which did not settle at all; various methods were tried to hasten the settling, but without effect, so it was finally abandoned.

C' (hydrochloric, cadmium in excess).—White precipitate which settled well, gave iron to cadmium 1:1.46 1:1.44. Results very similar to those obtained from A, and agreeing with a ratio of Fe:K: Cd of 7:8:10.

D' (hydrochloric, ferrocyanide in excess).—A greenish precipitate which would not settle. Separation by a centrifugal was tried as well as other methods, such as the addition of sodium chloride but without effect; finally it became so decomposed as to be worthless.

E' (acetic, cadmium in excess).—White, settled well, leaving a turbid solution; gave, on analysis, Fe:K: Cd :: 1:1.078: 1.383.

F' (acetic, ferrocyanide in excess).—This precipitate became very green on standing, and was rejected.

G' (ammonia, cadmium in excess).—A perfectly white curdy precipitate which settles immediately, leaving a clear colourless liquid, and differs entirely in physical properties from the preceding. It gave, on analysis, the ratio 1:0.227, showing, as in G, no potassium, and the presence of cadmium in excess of the possible ratio for a ferrocyanide.

H' (ammonia, ferrocyanide in excess).—A creamy white precipitate which settles well and has the consistency of clay, requiring considerable force to stir it up, and differing entirely in physical properties from the others. It gave, on analysis, Fe:K: Cd :: 1:2.07: 1.048 or nearly 1:2:1, corresponding to $\text{K}_2\text{CdFe}(\text{CN})_6$.

It remained to obtain precipitates to take the places of B', D', and F'. So in March, 1901, these precipitates were made again under the conditions already described, but in dark brown glass bottles, and were kept in the dark. This greatly lessened the decomposition, and by sacrificing a large proportion of the precipitates with the wash-water by July, residues were obtained which were free from excess of ferrocyanide, and only slightly bluish in colour.

B'' (neutral, ferrocyanide in excess).—Ratio, 1:1.81: 1.12, agreeing fairly with B and with $\text{K}_{18}\text{Cd}_{11}[\text{Fe}(\text{CN})_6]_{10}$.

D'' (hydrochloric, ferrocyanide in excess).—Ratio, 1:2.02: 0.955, confirming D, and corresponding to $\text{K}_2\text{CdFe}(\text{CN})_6$.

F'' (acetic, ferrocyanide in excess).—Ratio, 1:2.03: 0.95, confirming F, and corresponding to $\text{K}_2\text{CdFe}(\text{CN})_6$.

The abnormal results in G and G', probably due to cadmium hydroxide being carried down in varying quantities, necessitated a repetition of this work, so in July, 1901, G''' was made under the same conditions, but was treated repeatedly with very large quantities of ammonia before washing with water, and then analysed at once.

G''' (ammonia, cadmium in excess).—The iron cadmium ratio was determined in duplicate, and found to be 1:1.496, corresponding to $\text{K}_2\text{Cd}_3[\text{Fe}(\text{CN})_6]_2$.

* Washed six times with ammonia and water, then with water till free from ferrocyanide.

When this precipitate was being washed it was noticed that there was a portion which did not settle as well as the rest; this was decanted from the part which settled well, washed, and analysed, giving a ratio of exactly 1Fe: 1Cd.

In the results already given the duplicates on C' and the ratios on E and E' did not check satisfactorily, so these precipitates were made again (July, 1901), and as they settled well (cadmium being in excess) they were washed and analysed within a week.

C''' (hydrochloric, cadmium in excess).—White, no decomposition; gave Fe: Cd :: 1:1.07 and 1:1.065.

E''' (acetic, cadmium in excess).—White, no decomposition; gave Fe: Cd :: 1:1.07 and 1:1.06.

We see in these two a total lack of agreement in composition with those which were allowed to stand many months before analysis, indicating a change in composition after precipitation. On the other hand, these last results are in exact agreement with the titration ratios in slightly acid solution (Miller and Fisher, *Journ. Amer. Chem. Soc.*, 1900, xxi., 542); for if the theory for $\text{K}_4\text{Fe}(\text{CN})_6$ or 1Fe: 1Cd* gives for the strength of the ferrocyanide solution 1 c.c. = 0.00671 gm. cadmium, then, if the ratio in the precipitate is 1:1.07, the strength of the solution in terms of cadmium becomes 1.07 x 0.00671 or 0.00718, while the average result of titration was 0.00717.

For ease of comparison let us disregard for the present the potassium, which has been found to be present always in the proper amount to satisfy the remaining valency of the ferrocyanogen radical, and consider only the iron cadmium ratios.

Character of solution.	Cadmium in excess. Fe: Cd.	Ferrocyanide in excess. Fe: Cd.
Neutral	1:1.42 A	1:1.08 B
	1:1.448 A'	1:1.12 B''
	1:1.462 C'	1:0.99 D
	1:1.44 C'	1:0.955 D''
Hydrochloric ..	1:1.07 C''' (a)	—
	1:1.065 C''' (a)	—
	1:1.392 E	1:0.95 F
	1:1.383 E'	1:0.95 F''
Acetic	1:1.07 E''' (a)	—
	1:1.06 E''' (a)	—
	1:3.33 G	1:0.97 H
	1:1.91 G (b)	1:1.048 H'
Ammonia ..	1:1.94 G'	—
	1:2.27 G'	—
	1:1.496 G'''	—
	1:1.496 G'''	—
	1:1 G''' (c)	—
	1:1.496 G'''	—

(a) Analysed within a week of precipitation.

(b) After treatment with ammonia.

(c) Decanted portion.

We see from the work described that:—

1. The results when ferrocyanide is in excess are entirely different from those where cadmium is in excess.

2. In either acid or ammoniacal solution, ferrocyanide being in excess, the ratio is 1 iron to 1 cadmium, or the precipitate is $\text{K}_2\text{CdFe}(\text{CN})_6$, while in a neutral solution the cadmium ratio is higher.

3. With cadmium in excess, in either neutral or hydrochloric acid solution, the final composition is the same, corresponding to $\text{K}_8\text{Cd}_{10}[\text{Fe}(\text{CN})_6]_7$, while in an acetic acid solution the final composition corresponds to $\text{K}_6\text{Cd}_7[\text{Fe}(\text{CN})_6]_5$.

4. With cadmium in excess, in an acid solution the composition alters on standing with an increase of cadmium in the ratio, but when freshly precipitated the ratio agrees exactly with the results by titration.

5. With cadmium in excess, in an ammoniacal solution the ratio exceeds the normal, but after washing with am-

* Based on Hermann's formula, $\text{K}_2\text{CdFe}(\text{CN})_6$.

monia corresponds to $\text{Cd}_2\text{Fe}(\text{CN})_6$; when freshly precipitated and washed quickly with ammonia, the part which settles badly being removed by decantation, the composition agrees exactly with $\text{K}_2\text{Cd}_2[\text{Fe}(\text{CN})_6]_2$, while the part decanted corresponds to $\text{K}_2\text{CdFe}(\text{CN})_6$.

6. The constancy of the final composition of these precipitates does not favour the theory that potassium ferrocyanide is merely dragged down.

The results obtained when cadmium was in excess in an ammoniacal solution were so extraordinary that they deserved further study. The precipitate G'' , $\text{K}_2\text{Cd}_3[\text{Fe}(\text{CN})_6]_2$, was treated seven times with strong ammonia (sp. gr. 0.90) to see whether a change could be effected by a difference in solubility in ammonia, as the $\text{K}_2\text{CdFe}(\text{CN})_6$ seemed the more soluble. In this way, a creamy white residue was obtained which gave, on analysis, $\text{Fe} : \text{Cd} :: 1 : 1.99$, showing $\text{Cd}_2\text{Fe}(\text{CN})_6$, which was also obtained in G after re-washing with ammonia.

This result, together with the fact that the portion first obtained by decantation when the precipitate was originally treated with ammonia, gave a ratio of $\text{Fe} : \text{Cd}$ of exactly 1:1 shows that the original precipitate can be resolved into the two simple ferrocyanides $\text{Cd}_2\text{Fe}(\text{CN})_6$ and $\text{K}_2\text{CdFe}(\text{CN})_6$, and affords important confirmation of the theory that these complicated precipitates are mixtures, advanced several years ago in connection with the ferrocyanides of zinc and manganese (*Journ. Amer. Chem. Soc.*, 1897, xix., 556).

Now, if this is true for the precipitate in an ammoniacal solution, it ought also to be true of those more complicated ones formed in acid solutions; accordingly, C''' , which gave 1:1.07 when freshly precipitated, was treated in the same way with strong ammonia, and the residual cream-coloured portion analysed. This gave a ratio of 1:1.90, showing that the same change had taken place, though it was not entirely complete.

To test this further, precipitate A (neutral, cadmium in excess, made June, 1900) was treated eight times with strong ammonia and the residue analysed. This gave the ratio of 1:1.99, agreeing almost perfectly with $\text{Cd}_2\text{Fe}(\text{CN})_6$.

In order to ascertain whether the change which takes place on standing can be hastened by heating, the precipitate E''' (acetic, cadmium in excess), which when washed quickly and analysed gave a ratio of 1:1.07, was heated with water containing acetic acid on a water-bath for three days, and then analysed. The result was $\text{Fe} : \text{Cd} :: 1 : 1.40$ compared with 1:1.383 (E') after standing six months in the cold and 1:1.392 (E) after one year.

This precipitate (E''' after heating) was next treated eight times with strong ammonia, and the residual portion analysed. The result was $\text{Fe} : \text{Cd} :: 1 : 1.97$, showing again the presence of the normal cadmium ferrocyanide.

These experiments show that the ferrocyanides undergo a change in composition after precipitation, which progresses to the same point under the same conditions, and which is hastened by heating. The action of ammonia on these precipitates (cadmium in excess) has been confirmatory of the idea that they are either mixtures or else very easily decomposable double salts made up of $\text{K}_2\text{CdFe}(\text{CN})_6$ and $\text{Cd}_2\text{Fe}(\text{CN})_6$.

Written in this way the results are expressed as follows:—

Character of Solution : Neutral.

Cadmium in excess .. $4\text{K}_2\text{CdFe}(\text{CN})_6 \cdot 3\text{Cd}_2\text{Fe}(\text{CN})_6$
Ferrocyanide in excess $9\text{K}_2\text{CdFe}(\text{CN})_6 \cdot \text{Cd}_2\text{Fe}(\text{CN})_6$

Character of Solution : Hydrochloric.

Cadmium in excess .. $14\text{K}_2\text{CdFe}(\text{CN})_6 \cdot \text{Cd}_2\text{Fe}(\text{CN})_6$ (a)
Ferrocyanide in excess $\text{K}_2\text{CdFe}(\text{CN})_6$
Cadmium in excess .. $4\text{K}_2\text{CdFe}(\text{CN})_6 \cdot 3\text{Cd}_2\text{Fe}(\text{CN})_6$

Character of Solution : Acetic.

Cadmium in excess .. $14\text{K}_2\text{CdFe}(\text{CN})_6 \cdot \text{Cd}_2\text{Fe}(\text{CN})_6$ (a)
Ferrocyanide in excess $\text{K}_2\text{CdFe}(\text{CN})_6$
Cadmium in excess .. $3\text{K}_2\text{CdFe}(\text{CN})_6 \cdot 2\text{Cd}_2\text{Fe}(\text{CN})_6$

Character of Solution : Ammonia.

Cadmium in excess .. $\text{K}_2\text{CdFe}(\text{CN})_6 \cdot \text{Cd}_2\text{Fe}(\text{CN})_6$ (a)
Ferrocyanide in excess $\text{K}_2\text{CdFe}(\text{CN})_6$
(a) Freshly precipitated.

It would be premature to advance these formulæ as representing relations of these complicated and variable precipitates, but the facts seem to warrant this as a working hypothesis, to be used in a further study of the other insoluble ferrocyanides.

Although the results are not all that are desired, many of the precipitates are so easily oxidised, readily decomposed, and impossible to filter, that it seemed very doubtful whether any better results could be obtained if the work were repeated.

On comparing the results obtained on cadmium with those on zinc previously published (*Journ. Amer. Chem. Soc.*, 1897, xix., 547), and with those of Stone and Van Ingen (*Journ. Amer. Chem. Soc.*, 1897, xix., 542) we see some cases of remarkable agreement as well as some discrepancies, but a discussion of these results is postponed, as it is proposed to continue this work by a similar investigation of the ferrocyanides of the other metals of the periodic group containing cadmium and zinc, and also of the analytical group containing zinc, manganese, nickel, and cobalt, so that, taking zinc as a starting-point, we can see whether the variations in composition have any connection with the analytical grouping or the periodic law, and also possibly obtain more knowledge of their constitution.

THE CLASSIFICATION OF THE ELEMENTS.*

By HENRY E. ARMSTRONG, V.P.R.S.

(Concluded from p. 88).

It is unnecessary to go into further details—the table is self-explanatory; but it may not be superfluous to point out that the characteristic and most remarkable feature brought out by this mode of classifying the elements is the existence in various parts of the system of groups or series of related elements from the highest term of which alone "progression" takes place. The existence of such groups in the case of the iron and platinum metals and some of the rare earths, has long been recognised—but not their significance. It is not unlikely that some of the columns will be "cleared" of such groups when atomic weights generally are known with a closer approach to certainty. Of their existence in column 4, there can be no doubt; but if in column 7 rubidium were put at 84, 85 could be transferred to column 8; and if lower down in the same column, caesium were put at 131, 132 and 133 could be transferred to column 8, or the necessary "correction" might be made perhaps with greater advantage by including in column 4 two terms (81 and 82) in the fifth, and three (128, 129, 130) in the seventh period: column 7 would then be free from "grouped" elements. Such groups, however, are undoubtedly as characteristic of column 8 as of column 4; whether they are of column 9 is open to question—the cerium group might well follow barium in column 8; but wherever they may come, it is clear that the elements of this group are a very numerous body, and that a remarkable expansion may be looked forward to in this part of the table. Column 11 might also, by a similar process, be cleared of grouped elements. If such a clearance turn out to be possible, grouped elements will be characteristic of only three of the families—those in columns 4, 8, and 12.

Making allowances in the manner suggested, the "smoothed" scheme is arrived at which is embodied in Table II.

The possibilities disclosed by a system of classification such as that here suggested are remarkable, but they are on the surface and need not be dwelt upon. Speculation

* A Paper read before the Royal Society, March 20, 1902.

TABLE II.

1 H 17 33	2 He 18 34	3 Li 19 F 35 Cl	4 Be 20 A 36	5 B 21 37	6 C 22 38	7 N 23 Na 39 K	8 O 24 Mg 40 Ca	9 Ne 25 41	10 Ar 26 42 Kr	11 B 27 Al 43 Sb	12 C 28 Si 44 45 46 47 Ti 48	13 N 29	14 O 30	15 F 31 P	16 S 32 S
53	54	55 Mn	56 Fe 57 58 59 Co Ni	60	61	62	63 Cu 64 65 Zn 66	67	68 X	69 Ga	70 71 72 Ge 73	74	75 As	76	77 Se
78	79	80 Br	81 82	83	84	85 Rb	86 87 Sr	88	89	90 Yt	91 Zr 92	93	94	95 Nb	96 Mo
97	98	99	100 101 102 Ru 103 Rh 104 105 Pd	106	107	108 Ag	109 110 111 112 Od	113	114	115 In	116 117 Vc (?) 118 Sn 119 120	121 Sb	122	123	124 Te
125	126	127 I	128 129 130	131	132	133 Os	134 135 136 Ba 137 La 138 139 140 Ce 141 Pr 142 143 144 Nd 145 146 147 148 149 150 Sm								

Y51 En

185	186	187	188 189 190 191 Os 192 193 Ir 194 Pt	195	196	197 Au	198 199 200 Hg 201	171	172	173 Yb	174 175 176 177 178 179 180	181	182	183 Ta	184 W
212	213	214	215 216 217 218 219 220	221	222	223	224 225 226 227 228	202	203	204 Tl	205 206 207 Pb	208 Bi	209	210	211
								229	230	231	232 Th 233 234 235 236	237	238	239	240 U

on such a subject will be justified if it but lead to further appreciation of the rhythm which undoubtedly underlies the relationship subsisting among the elements. That work in plenty is left for the chemist to do is certain.

THE VOLUMETRIC ESTIMATION OF ALUMINA, AND FREE AND COMBINED SULPHURIC ACID IN ALUMS.*

By ALFRED H. WHITE.

In judging the quality of an alum, among the important determinations are those which give the amount of soluble alumina and the amount of sulphuric acid in combination with it or existing as free acid. The alumina may be satisfactorily estimated gravimetrically, but the method is tedious. A gravimetric estimation of the sulphuric acid gives not only that combined with aluminum plus that present as free sulphuric acid, but also that present as sodium sulphate, &c., and the amount of alkalis must be known before the amount of sulphuric acid combined with aluminum can be determined.

The determination of free sulphuric acid in alums by volumetric means has been repeatedly attempted. The hydrolysis of aluminum sulphate prevents direct titration with an alkali, since as fast as the free acid present is neutralised, more is formed by hydrolysis, so that the solution will not remain neutral for an appreciable length of time until nearly all the acid originally combined with the aluminum has been neutralised, and most of the aluminum hydroxide precipitated. This cannot be made into a practicable quantitative method for the estimation of free and combined acid, as the formation of aluminate with alkaline reaction begins before all the hydroxide has been precipitated. Further, if phenolphthalein is used as indicator, the precipitated hydroxide obstinately holds, by adsorption, the pink colour even when the solution is no longer alkaline, so that the method—though perhaps giving in the hands of works chemists in constant practice results which are fairly concordant—is, because of these various errors, not to be considered a practicable analytical method. The following paper results from an attempt to work out a practicable method.

A standard solution of barium hydroxide was used instead of caustic soda, to avoid any trouble caused by carbon dioxide in the caustic affecting the phenolphthalein used as indicator. It was found later that the barium hydroxide possessed other advantages. To prevent the adsorption of pink colour by the precipitated aluminum hydroxide obscuring the end-reaction, an addition of neutral potassium-sodium tartrate (Rochelle salt) was made to hold the alumina in solution. The modification proved a good one. Duplicate results checked closely. There was no precipitation of alumina during the titration, nor even of barium sulphate. The solution remained perfectly clear and colourless until the end-reaction, which was sharp. After standing a short time the solution became opalescent, and then milky, but no precipitate settled out. This marked retardation of the precipitation of barium sulphate was unexpected, and the conditions under which it occurs are undergoing further investigation.

To determine that the results thus obtained were accurate, a solution of aluminum sulphate was made by precipitating aluminum hydroxide from a solution of aluminum chloride with ammonia, washing the precipitate thoroughly, and then dissolving in standard sulphuric acid, which was afterward diluted to a litre (Solution A). The solution thus obtained was almost fifth normal, and the amount of acid was slightly in excess of the amount theoretically necessary to form the normal aluminum sulphate. A duplicate solution of acid

alone, carefully standardised gravimetrically, was used to check the results.

The method of procedure was as follows:—To 25 c.c. of the fifth-normal alum solution was added 50 c.c. of 10 per cent neutral potassium-sodium tartrate, and the mixture was titrated cold with barium hydroxide solution, using phenolphthalein as indicator. This amount of tartrate was sufficient to prevent anything more than a slight opalescence in the solution before the end-reaction. Duplicate titrations required 25.85 and 25.80 c.c. of fifth-normal barium hydroxide. To determine the effect of the amount of tartrate, another titration was made, using 100 c.c. of tartrate instead of 50. The amount of barium hydroxide solution used was 25.85, as before. The check solution of sulphuric acid required 25.65 c.c. barium hydroxide, no difference being apparent whether no tartrate, 50 c.c., or 100 c.c. of tartrate were present. The solution containing the aluminum sulphate therefore requires slightly more barium hydroxide than that containing the sulphuric acid alone. A possible explanation is that the ionisation of the barium hydroxide is lessened to such an extent by the aluminum tartrate complex that it requires an appreciably large excess of barium hydroxide to bring about the end reaction. If, however, the barium hydroxide is standardised against a solution of aluminum sulphate made from precipitated alumina and sulphuric acid as described, the results will be constant and give accurate results when extended to other alums. The addition of sodium sulphate in amount equivalent to the amount of sulphuric acid combined with the aluminum does not affect the result; the addition of standard sulphuric acid increases the amount of barium hydroxide used by the theoretical amount. The addition of neutral tartrate and titration with barium hydroxide, therefore, affords an accurate method of determining the total sulphuric acid combined with the aluminum plus the excess of free acid, irrespective of the amount of sulphuric acid combined with alkalis.

As it is well known that the hydroxy-organic acids in general have the power of preventing the precipitation of alumina, salts of other acids than tartaric were tried, among them neutral sodium citrate. Sodium citrate prevented the precipitation of alumina, retarded the precipitation of barium sulphate, and allowed a perfectly sharp end-reaction, as did the tartrate, but the amount of barium hydroxide used was only a little over two-thirds of that required when titrating in the presence of the tartrate. Solution A, with neutral sodium citrate added, required only 17.95 c.c. barium hydroxide instead of 25.84 when tartrate was used. Experiment showed that the addition of sodium sulphate did not affect the result, and that an addition of standard sulphuric acid caused the theoretically calculated increase in the barium hydroxide used. It seemed that the result obtained when titrating in the presence of citrate could be due only to the formation of a complex aluminum ion, and that this might furnish the basis of a method for the estimation of the aluminum. If we assume that the barium hydroxide used when titrating in presence of tartrate, represents free acid plus acid combined with alumina, while the barium hydroxide used when titrating in presence of citrate represents free acid plus two-thirds of acid combined with alumina, the difference represents one-third of the sulphuric acid combined with the alumina, or one-third the alumina. In the above instance, $25.84 - 17.95 = 7.89$ c.c. fifth-normal barium hydroxide, and calculating the alumina on the assumption that this is equivalent to one-third of it, we find that we get 0.0805 gm. of alumina as compared with 0.0831 gm. obtained gravimetrically. The volumetric result is too low. It seemed entirely possible that partial hydrolysis of the alum in the citrate solution might cause more barium hydroxide to be used than called for by the above supposition, and that this might account for the low result. Accordingly, among other variations, the solution of aluminum sulphate was evaporated to dryness on the water-bath and dissolved in 50 c.c. of 10 per cent sodium citrate

* Contribution from the Chemical Laboratory of the University of Michigan. From the *Journal of the American Chemical Society*, xiv., No. 5.

and titrated. There were required only 17.58 c.c. barium hydroxide instead of 17.95 c.c., and the alumina calculated from this is 0.0842 grm. instead of 0.0831 grm. obtained gravimetrically. Using a saturated solution of citrate to re-dissolve the alum did not give an appreciably different result. Thus, the first method gives results considerably low, while the second gives slightly higher results. Some further experiments upon the influence of concentration and time follow, made upon a C. P. aluminum sulphate in a solution of 30 grms. per litre (Solution B).

B.	Volume aluminum sulphate.	Water added.	Citrate added.	Barium hydroxide.	Remarks.
1.	25	0	50	21.75	Titrated at once.
2.	25	25	50	21.76	Titrated at once.
3.	25	50	50	21.67	Water added; stood fifteen minutes; then citrate added, and titrated.
4.	25	100	50	21.70	Water added; stood fifteen minutes; then citrate added, and titrated.
5.	25	200	50	21.72	Water added; stood fifteen minutes; then citrate added, and titrated.
6.	25	Evaporated to dryness and re-dissolved in 50 c.c. 10 per cent citrate; stood ten minutes before titrating; required 21.62 c.c. barium hydroxide.			
7.	0.750 grm. (25 c.c.) of solid salt, dissolved in 50 c.c. 10 per cent citrate and titrated at once, required 21.90 c.c. barium hydroxide.				

The above series of experiments shows that the addition of varying amounts of water and variation of time within short intervals makes but slight difference in the result. The hydrolysis is apparently a slow one. In twenty-four hours, however, equilibrium is practically complete, as is shown by another series of experiments on a commercial aluminum sulphate dissolved to a strength of 30 grms. per litre (Solution C).

C.	Volume aluminum sulphate.	20 per cent citrate added.	Barium hydroxide.	Remarks.
1.	25	25	21'0	Titrated at once.
2.	25	25	21'02	Stood fifteen minutes before titrating.
3.	25	25	20'62	Stood sixteen hours before titrating.
4.	25	Evaporated to dry- ness and re-dis- solved in 50 c.c. 10 per cent ci- trate.	20'53	Titrated at once.
5.	25	Evaporated to dry- ness and re-dis- solved in 50 c.c. 10 per cent ci- trate.	20'69	Stood ten minutes be- fore titrating.
6.	0.750 grm. (25 c.c.) solid salt dissolved in 50 c.c. 10 per cent citrate.		21'10	Stood ten minutes be- fore titrating.

Nos. 3 and 5 show that if sufficient time is given to allow equilibrium to be established, the results are practically the same whether the 50 c.c. solution is evaporated to dryness and dissolved in 50 c.c. of 10 per cent citrate (20.69 c.c.), or 25 c.c. of solution are treated with 25 c.c. of 20 per cent citrate, and the solution allowed to stand sixteen hours (20.62 c.c.). No. 6 in this series, as well as No. 7 in Series B, shows that the same result is not reached when dissolving the solid

salt in citrate (B 7 = 21.90) and titrating as when evaporating to dryness, and re-dissolving in the same amount of citrate (B 6 = 21.62). Commercial alum is evidently not a homogeneous body, and further combination between the alumina or basic sulphate and the sulphuric acid takes place after solution in water.

The analytical method as finally worked out is as follows:—Dissolve 3 grms. of alum in 100 c.c. of water. Take 25 c.c. sample, add 50 c.c. strictly neutral 10 per cent potassium-sodium tartrate, and titrate with fifth-normal barium hydroxide, using phenolphthalein as indicator. This is equivalent to the sulphuric acid combined with the alumina plus the free acid. Evaporate a duplicate 25 c.c. sample to dryness on the water-bath, dissolve in 50 c.c. strictly neutral 10 per cent sodium citrate, allow to stand ten minutes, and titrate with barium hydroxide, with phenolphthalein indicator as before. The difference between these results is equivalent to one-third of the sulphuric acid combined with the alumina, and hence to one-third of the alumina. The barium hydroxide solution should be standardised by a blank determination upon a solution of sulphuric acid in which approximately enough precipitated aluminum hydroxide has been dissolved to correspond to aluminum sulphate. The aluminum hydroxide may be best made by precipitation of the chloride to ensure absence of sulphate. Caustic soda, even when freed from carbon dioxide by barium hydroxide, does not give such satisfactory results as the barium hydroxide. As examples of the practicability of the method the following results on two widely differing alums are cited:—

Alum B, a C.P. Aluminum Sulphate.

30 grms. per litre (25 c.c.) sample = 0.750 grm. sample.

	C.c.
Fifth-normal barium hydroxide for tartrate titration	32.50 32.55
Fifth-normal barium hydroxide for citrate titration	21.60 21.63
Difference	10.90

10.90 × 3 = 32.70 c.c. fifth-normal barium hydroxide equals total sulphur trioxide theoretically necessary to combine with alumina, and therefore equals alumina.

32.70 × 0.003407 = 0.1114 grm. alumina equals 14.86 per cent alumina. Alumina determined gravimetrically equals 14.73 and 14.80.

The close agreement between the figures 32.52 c.c. (free and combined sulphur trioxide) and 32.70 c.c. (combined sulphur trioxide) show that the alum is almost exactly neutral with an excess equal to 0.18 c.c. of fifth-normal alumina.

Alum C, a Commercial Aluminum Sulphate.

30 grms. per litre (25 c.c.) sample = 0.750 grm. sample.

	C.c.
Fifth-normal barium hydroxide for tartrate titration	33.59 33.59
Fifth-normal barium hydroxide for citrate titration	20.64 20.74
Difference	12.90

12.90 × 3 = 38.70 c.c. fifth-normal barium hydroxide equals total sulphur trioxide theoretically necessary to combine with alumina, and therefore equals alumina.

38.70 × 0.003407 = 0.1318 grm. alumina equals 17.58 per cent alumina. Alumina determined gravimetrically equals 17.57 and 17.50.

38.70 - 33.59 = 5.11 c.c. fifth normal alumina equals 0.0174 grm. alumina equals 2.32 per cent alumina more than is sufficient to form aluminum sulphate.

Alum B, as shown by the above figures, is slightly basic, but a determination of free acid by the method of

Beilstein and Grosset gave 0.9 per cent free acid (*Bulletin de l'Académie Impériale des Sciences in St. Petersburg*, 1890, p. 147). This method of Beilstein and Grosset has been investigated by V. Keler and Lunge (*Zelt. Angew. Chem.*, 1894, p. 669), who state that it gives results which are uniformly slightly high but otherwise very accurate, and it therefore became necessary to explain the discrepancy. The method of Beilstein and Grosset is as follows:—Dissolve 1 or 2 grms. of alum in 5 c.c. of cold water, add 5 c.c. of a cold saturated solution of ammonium sulphate, allow to stand, with frequent stirring, for fifteen minutes, and then add 50 c.c. 95 per cent alcohol. Filter, wash with 50 c.c. 95 per cent alcohol, evaporate to dryness on water-bath, dissolve in water, and titrate with tenth-normal potassium hydroxide, using litmus as indicator. All the alum is, in this process, supposed to be precipitated as normal ammonium alum together with most of the excess ammonium sulphate, and the free sulphuric acid remains in solution together with a small amount of ammonium sulphate. Three determinations of free acid in alum B by this method gave 0.86, 0.88, and 0.89 per cent free acid as sulphur trioxide—as good an agreement as could be desired. To test whether an error in Beilstein and Grosset's method due to hydrolysis might not be responsible for this apparent free acid, variations were made in amount of water and concentration of sulphur trioxide with the following results:—

Sample Grms.	Water. C.c.	Saturated solution (NH ₄) ₂ SO ₄ . C.c.	Alcohol. Per cent.	Free acid as SO ₃ . Per cent.
2	10	10	95	0.96
3	10	10	95	0.88
3	5	10	absolute	0.70
3	5	5+5 gm. solid ammonium sulphate.	absolute	0.61

Blank determinations showed that the change in results was not due to impurities in the ammonium sulphate or alcohol. The results show that the decrease in the relative amount of water decreases the apparent amount free acid, but although hydrolysis does thus play a part in influencing the results, there remains as the lowest figure, 0.6 per cent, which can hardly be attributed to an error in the method. Turning back to the series of results obtained by titrating alum B in presence of citrate, it was remarked that the figures obtained by dissolving the alum directly in citrate were higher than those obtained by dissolving the alum in water, then evaporating to dryness, and re-dissolving in citrate which showed a greater amount of free acid in the original alum than after evaporation to dryness. If the results in B 7 are taken and calculation made we find that there are required:—

Fifth-normal barium hydroxide for tartrate titration	C.c.
Fifth-normal barium hydroxide for citrate titration when alum dissolved directly in citrate	32.52
Difference	21.90
10.62 x 3 = 31.86 c.c.	
32.52 - 31.86 = 0.66 c.c. = 0.70 per cent free acid as sulphur trioxide.	

This result is in fair accord with the results obtained by the Beilstein and Grosset method of direct estimation, and probably corresponds closely to the amount of free acid actually present in the solid salt. It is entirely incorrect, however, to hold that the solution of this alum will have that amount of free acid, as the results already given show that the amount of alumina in solution is slightly greater than the amount necessary to form the normal salt. The solid salt is evidently not uniform. When dissolved in water to dilute solution, these inequalities disappear quickly. In concentrated solution, as in the Beilstein and Grosset method, or when the salt is dissolved in citrate, equilibrium is attained much more slowly, and a titration

of such a solution soon after solution is complete gives approximately the conditions prevailing in the solid salt. If the solution is allowed to stand a sufficient length of time, equilibrium is finally reached, as shown by experiments C 3 and C 5 (*ante*).

Summary.

If a solution of an alum to which has been added neutral potassium sodium tartrate (Rochelle salt) is titrated with barium hydroxide, the barium hydroxide used will correspond to the sulphuric acid combined with the alumina plus the free acid. The sulphuric acid combined with sodium or potassium is not estimated. If a duplicate solution of alum is evaporated to dryness, re-dissolved in neutral sodium citrate, and titrated with barium hydroxide, a smaller quantity of barium hydroxide is required, and the difference between the amounts of barium hydroxide used in the two titrations is equivalent to one-third of the alumina. From these two titrations can be calculated the alumina and the sulphuric acid combined with it, whether the alum be basic or acid, and if the alum is acid, the excess of acid over that necessary to form the normal sulphate. Commercial aluminium sulphate may, in its solid state, carry free acid, although in the solution such uncombined acid may disappear, combining with what had been basic portions of the solid salt. Such free acid may be estimated by dissolving the solid salt directly in citrate, and titrating with barium hydroxide at once. This method gives results closely concordant with Beilstein and Grosset's method, but it does not show that the alum contains more acid than is sufficient to form with the alumina the normal salt.

When aluminium sulphate is titrated with barium hydroxide thus, in presence of neutral alkali tartrate or citrate, the precipitation of barium sulphate is retarded for a time, varying from a few minutes to several hours, and when the precipitate does form it is in a very peculiar colloidal form which is undergoing further investigation. Consideration of this subject, and also of the action of salts of other metals than aluminium and salts of other acids than tartaric and citric, as well as the theoretical points involved, are reserved for a following paper.

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ERRATUM.—P. 87, line 1, col. 1 of Table, for "1 Hg" read "1 H."

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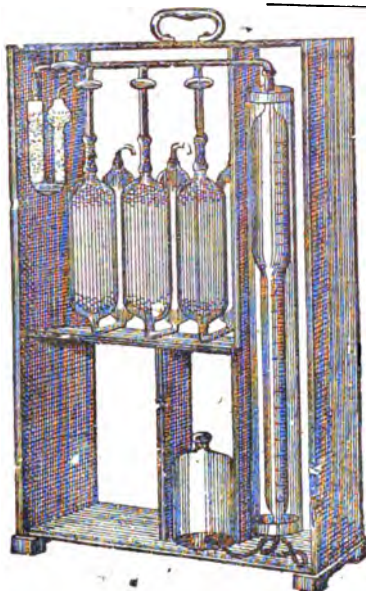
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UNIVERSITY COLLEGE, SHEFFIELD.

The Council invite Applications for the Post of **JUNIOR DEMONSTRATOR** in the **CHEMICAL LABORATORY**, at a salary of £100 per annum.

Applications to be sent in on or before **SEPTEMBER 17th** to the Professor of Chemistry, from whom further particulars may be obtained.

THE CHEMICAL NEWS.

Vol. LXXXVI., No. 2322.

(STUDENTS' NUMBER).

UNIVERSITIES AND COLLEGES.

UNIVERSITY OF LONDON.

CANDIDATES for any Degree in this University must either have passed the Matriculation Examination in this University, or be admitted under the Statute which provides that the Senate may admit graduates of or persons who have passed the examinations required for a degree in other Universities approved by it. This and all other Examinations of the University, together with the Prizes, Exhibitions, Scholarships, and Medals depending upon them, are open to Women upon exactly the same conditions as to Men.

There are three Examinations for Matriculation in each year; one commencing on September 15th, one on the second Monday in January, and the third on the second Monday in June (or July, as may hereafter be determined).

The Regulations for Matriculation have recently undergone revision, but an Examination under the old Regulations will be held in January, 1903, and (in addition to that under the revised Regulations) in June, 1903, but not thereafter.

The Examination is conducted by means of Printed Papers; but the Examiners are not precluded from putting, for the purpose of ascertaining the competence of the candidates to pass, *viva voce* questions to any candidate in the subjects in which they are appointed to examine. These Examinations (except that in September) may be held not only at the University of London, but also, under special arrangement, in other parts of the United Kingdom, or in the Colonies.

Every candidate for the Matriculation Examination must apply to the Principal for a Form of Entry, which must be returned within a specified time before the commencement of the Examination, accompanied by a Certificate showing that the candidate has completed his sixteenth year, and by his Fee for the Examination. As no candidate can be admitted after the List is closed, any candidate who may not have received a Form of Entry within a week after applying for it must communicate immediately with the Principal, stating the exact date of his application and the place where it was posted.

Every candidate entering for the Matriculation Examination must pay a Fee of £2. If a candidate withdraws his name, or fails to present himself at the Examination, or fails to pass it, the Fee shall not be returned to him.

Candidates are not approved by the Examiners unless they have shown a competent knowledge in each of the following five subjects:—(1) English; one paper of three hours. (2) Elementary Mathematics; two papers of three hours each. (3) Latin or Elementary Mechanics, or Elementary Physics—Heat, Light, and Sound, or Elementary Chemistry, or Elementary Botany; one paper of three hours in each subject. (4) and (5) Two of the following subjects, neither of which has already been taken under Section (3); one paper of three hours in each subject; if Latin be not taken, one of the other subjects selected must be another Language from the List, either ancient or modern:—Latin, Greek, French, German, Arabic, Sanskrit, Spanish, Portuguese, Italian, Hebrew, Ancient History, Modern History, Logic, Physical and General Geography, Geometrical and Mechanical Drawing, Mathematics (more advanced), Elementary Mechanics, Elementary Chemistry, Elementary Physics—Heat, Light, and Sound, Elementary Physics—Electricity and Magnetism, Elementary Biology

—Botany, Elementary Biology—Zoology. Candidates for Examination in subjects marked with an asterisk must give at least two months' notice; there will be no Examination in them in September, 1902.

A Pass Certificate, signed by the Principal, will be delivered to each successful candidate after the Report of the Examiners has been approved by the Senate.

Several valuable Scholarships and Exhibitions are available to students.

INTERMEDIATE EXAMINATION IN SCIENCE.

The Intermediate Examination in Science will commence on the second Monday in July.

No candidate (with the exception of such as have obtained Honours at the Matriculation Examination in the preceding January) is admitted to this Examination within one academical year of the time of his passing the Matriculation Examination.

The Fee for this Examination is £5.

Examination for Honours.

Candidates for Honours in Chemistry will be examined in Inorganic Chemistry, treated more fully than in the Pass Examination. This Examination will consist of two printed papers and a practical examination.

In the Examination for Honours, the Candidate, not being more than 22 years of age at the commencement of the Pass Examination, who most distinguishes himself will receive an Exhibition of £40 per annum for the next two years.

B.Sc. EXAMINATION.

The B.Sc. Examination will be held on the fourth Monday in October.

Candidates for this Examination are required to have passed the Intermediate Examination in Science at least one academical year previously, and to have passed the Matriculation Examination at least three years previously.

The Fee for this Examination is £5.

Examination for Honours.

For the examination for Honours in Chemistry two papers will be set and a practical examination.

The candidate, being not more than 23 years of age, who most distinguishes himself in Chemistry, will receive £50 per annum for the next two years, with the style of University Scholar.

DOCTOR OF SCIENCE.

The examination for the Degree of Doctor of Science takes place annually within the first twenty-one days of June.

No candidate is admitted to the examination for the Degree of D.Sc. until after the expiration of two Academical Years from the time of his obtaining the Degree of B.Sc. in this University.

Every candidate for this Degree must state in writing the special subject within the purview of the Faculty of Science, as set out in the Programme of the B.Sc. Examination, upon a knowledge of which he rests his qualification for the Doctorate; and with the Form of Entry he shall transmit an original Dissertation or Thesis (at least six copies), printed, type-written, or published in his own name, treating scientifically some special portion of the subject so stated, embodying the result of independent research, or showing evidence of his own work, whether conducted independently or under advice, and whether based on the discovery of new facts observed by himself, or of new relations of facts observed by others, or, generally, tending to the advancement of Science. Every candidate may further specify any printed contribution or contributions to the advancement of Science which he has at any time previously published. If the Dissertation or Thesis be approved by the Examiners, the candidate shall be required to present himself at the University upon such day or days within the first twenty-one days of June as may be notified to him, and shall, at the discretion of the Examiners, be further tested, either orally or practically, or by printed questions or by all of these methods, with reference both to the special subject selected by him and to the Thesis.

PRELIMINARY SCIENTIFIC (M.B.) EXAMINATION.

This Examination takes place twice in each year,—once, for Pass and Honours, commencing on the second Monday in July; and once for Pass Candidates only, commencing on the third Monday in January.

The Fee for this examination is Five Pounds.

UNIVERSITY OF OXFORD.

Waynflete Professor of Chemistry—W. Odling, F.R.S.
Less Reader in Chemistry—A. Vernon Harcourt, F.R.S.

Every Student must reside in one or other of the Colleges or Halls, or in licensed lodgings, for a period of three years. Students of Chemistry can obtain the degree of B.A. by passing preliminary examinations in Arts and in Science, and a final Honour examination in Chemistry. Chemistry may also be taken as part of the examination for a Pass degree. Graduates of other Universities suitably qualified can obtain the degree of Bachelor of Science after an approved course of study or research and two years' residence.

University Laboratory.—Demonstrators, W. W. Fisher, J. Watts, V. H. Valey, F.R.S., J. E. Marsh.—The fee for students working in the Laboratory for three days in the week during the Term is £3; for students working every day, £5.

Scholarships of about the value of £80 are obtainable at the majority of the colleges, by competitive examination in Natural Science.

More detailed information may be obtained from the Examination Statutes; the Student's Handbook to the University; and from the professors and college tutors.

UNIVERSITY OF CAMBRIDGE.

Professor of Chemistry—G. D. Liveing, M.A., F.R.S.
Jacksonian Professor of Natural and Experimental Philosophy—J. Dewar, M.A., F.R.S.

The Student must enter at one of the Colleges or Hostels, or as a Non-collegiate Student, and keep terms for three years by residence in the University. He must pass the previous examination in Classics and Mathematics, which may be done in any term of residence or before commencing residence. He may then proceed to take a Degree in Arts, either continuing mathematical and classical study, and passing the ordinary examinations for B.A., or going out in one of the Honour Triposes.

A graduate of another University may be admitted as an "advanced" student, and obtain a degree after two years' residence in the University.

The scholarships, ranging in value from £20 to £100 a year, are chiefly given for mathematical and classical proficiency. Scholarships, or Exhibitions, are given for Natural Science in King's, Trinity, St. John's, St. Peter's, Clare, Trinity Hall, Queen's, Jesus, Christ's, Emmanuel, Sidney, Pembroke, Caius, and Downing Colleges; the dates of the examinations vary, but are always fully advertised.

The Chemical Laboratory of the University is open daily for the use of the Students. The Demonstrators attend daily to give instructions. A list of the lectures is published annually, in June, in a special number of the *Cambridge University Reporter*, which may be had from the Cambridge Warehouse, in Paternoster Row, or through any bookseller.

Non-collegiate Students are allowed to attend certain of the College Lectures and all the Professors' Lectures, and have the same University status and privileges as the other Students. Full particulars may be obtained by forwarding a stamped directed envelope to the Assistant Registrar, Cambridge, or from the *Cambridge University Calendar*.

UNIVERSITY OF DUBLIN.

TRINITY COLLEGE.

Professor of Chemistry—J. Emerson Reynolds, D.Sc., M.D., F.R.S.

Assistant Lecturer—Emil A. Werner, F.C.S., F.I.C.

Demonstrators—W. Haughton, M.B., and W. C. Ramsden.

The general Laboratories include working accommodation for 120 Students, and the Quantitative and Research Laboratories for about 40 Students. The Laboratories will open on the 2nd of October. Lectures will commence about November 1st.

The Laboratories and the Lectures of the Professor of Chemistry can now be attended by Students who do not desire to reside in the University or proceed to its Degrees.

The full Course of General and Analytical Chemistry occupies three years, but a Student is free in his third year to devote most of his time to a special department of Pure or Technical Chemistry. Students can enter for any portion of the Course. The following Lectures are delivered:—

1. *Inorganic Chemistry and Chemical Philosophy*.—Elementary, first year; Advanced, including Physical Chemistry, second year.
2. *Organic Chemistry*.—General, second year; advanced, third year.
3. *Metallurgy*.—A Course for Engineering and Technical Students.

The Laboratories are open every day from 10 to 5 o'clock (except Saturdays, when they close at 1 o'clock).

The Summer Course of Practical Chemistry for Medical Students begins during the first week in April and terminates with the first week in July.

The University of Dublin grants the Degree of Doctor of Science to graduates of Master's standing whose independent researches in any branch of Science are of sufficient merit.

By recent decrees, Prizes in Chemistry and Physics will be given in future at the October (Arts) Entrance Examination, and also at the Terminal Examinations of the Junior and Senior Freshmen Years.

Similarly, two Science Scholarships will be obtainable by Freshmen, and tenable for five years. These Foundation Scholars receive £20 per annum, have free commons, their chambers for half the charge paid by other students, and their tutorial fees are at one-fourth the usual rates.

KING'S COLLEGE.

(DIVISION OF ENGINEERING, ARCHITECTURE, AND APPLIED SCIENCE).

Professor of Chemistry—J. M. Thomson, F.R.S., F.C.S.
Lecturer—Herbert Jackson, F.C.S.

Demonstrators—P. H. Kirkaldy, F.C.S., and D. Northall Laurie.

The Academical Year consists of Three terms. The days fixed for the Admission of New Students in the Academical Year 1902-1903 are October 2, January 15, and April 23.

Students of the First Year are admitted to the Course of Theoretical and Applied Chemistry. The Course commences with a view of the conditions suitable for the production of Chemical Phenomena, after which the laws of Chemical Attraction are discussed, and the Non-metallic Elements and their principal compounds are described. The Metals and their principal compounds are next examined, care being taken to point out the applications of the Science to the Arts; and the processes of the different Manufactures and of Domestic Economy are explained and illustrated. Examinations of the Class, both *visd voce* and by written papers, are held at intervals during the course at the usual Lecture hour.

Second Year.—Students attend in the Laboratory twice a week, and they go through a course of Chemical Analysis, both by liquid tests and the blowpipe, analysis of ores, liquids, &c., and Elementary Volumetric Analysis.

Experimental and Analytical Chemistry in the Laboratory.—The object of this Class is to afford to Students who are desirous of acquiring a knowledge of analysis, or of prosecuting original research, an opportunity of doing so under the superintendence of the Professor and Demonstrator; Students may enter, upon payment of extra fees, at any time except during the vacation, and for a period of one, three, six, or nine months, as may best suit

their convenience. The laboratory hours are from ten till four daily, except Saturday.

In addition to the Laboratory Fee, each Student defrays the expenses of his own experiments. The amount of this expense, which is comparatively trifling, is entirely under his own control.

Special hours and fees are arranged for the convenience of such Third Year Students as wish to study Analytical Chemistry.

Fees.—Chemistry per term, £3 3s.; per ann., £8 8s.; Practical Chemistry per term, £4 4s.; per ann., £10 10s.; Experimental and Analytical Chemistry—Five days a week: One month, £4 4s.; Three months, £10 10s.; Six months, £18 18s.; Nine months, £26 5s. Three days a week: One month, £2 12s. 6d.; Three mos., £6 6s.; Six mos., £11 11s.; Nine mos., £15 15s.

METALLURGY.

Professor—A. K. Huntington, F.I.C., F.C.S., &c.

The following subjects are treated of in the Lectures: The Selection and Economic Preparation of Fuel and of Refractory Materials; the methods by which metals are obtained from their ores, and the means by which they are rendered suitable for the various requirements of the Arts.

Particular attention is paid to the study of the Nature and Properties of Metals and Alloys available for Constructive Purposes.

In the Metallurgical Laboratory, which is always open during College hours, the relation between the Chemical Composition of Metals and their Mechanical Properties may be studied by the aid of Testing Machinery.

Fee, £3 3s. for the Course.

PHOTOGRAPHY.

Lecturer—Prof. J. M. Thomson, F.R.S., F.C.S.

In addition to the regular College Course in Photography occasional classes may be formed. For further particulars application should be made to Prof. Thomson.

EVENING CLASSES.

Classes for Evening Instruction in various subjects are held during the Winter Session.

UNIVERSITY COLLEGE.

FACULTY OF SCIENCE.

Professors—Sir William Ramsay, LL.D., F.R.S., &c. (Inorganic and Physical Chemistry); J. Norman Collie, Ph.D., F.R.S. (Organic Chemistry).

Assistants—Morris Travers, D.Sc.; E. C. C. Baly, F.I.C.; and F. G. Donnan, M.A., Ph.D.

The Session is divided into three Terms, as follows:—First Term, from Thursday, October 2nd, till Friday, December 19th; Second Term, from Tuesday, January 13th, till Friday, March 27th; Third Term, from Tuesday, April 21st, till Saturday, June 13th. Class Examinations begin on June 15th.

Junior Course of Inorganic Chemistry.

Tuesday and Thursday, at 9. Practical, Saturday, 11 to 1. Fee, £10 10s.

This Course treats of the chief physical and chemical characters of the Non-metallic Elements, their preparation and characteristic tests.

Senior Course of Inorganic Chemistry.

First and Second Terms: The Class meets four times a week, on Mondays, Wednesdays, Fridays, and Saturdays, at 9, for Lectures, Examinations, and Exercises.

Fees:—For the Course, £7 7s.; Perpetual, £9 9s.; for the First or Second Term, £4 4s.

In this Course the physical principles bearing on Chemistry, are first discussed, and the Chemistry of the Elements and their compounds are treated in considerable detail, under the headings—Elements; halides, oxides, sulphides, &c., borides, carbides and allicides, nitrides, phosphides, &c., and alloys. Special attention is devoted to substances of commercial importance. The concluding Lectures are devoted to Physical Methods and their bearing on Chemical Problems.

Third Term: Lectures will be given on Mondays and Fridays at 9 o'clock, on the chief types of Carbon Compounds.

An Exercise Class will meet once a week throughout the Session.

Advanced Course of Chemistry.

Second and Third Terms.—The class meets twice a week, on Tuesdays and Thursdays, at 9, beginning in January. The hour will be altered by special arrangement with the class if necessary.

Fee:—For the Course, £3 3s.; for a Term, £2 2s.

The Principles of Chemistry will be specially considered, comprising the Atomic Theory, Chemical Classification, the Periodic Law, Thermal Chemistry, Dissociation, the Application of Physical Methods to the Solution of Chemical Problems, and the influence of Mass and Temperature on the progress of Reactions. Special attention is paid to the most recent researches bearing on Chemical Theory.

Third Term: Some Lectures will also be given on Saturdays at 9, during the Third Term, on the History of Chemistry.

Organic Chemistry.

Mondays, Wednesdays, and Fridays, at 9, during the session.

This Course will consist of three lectures a week during the whole Session, and is suitable for Students who intend to study Chemistry from a scientific standpoint. No previous knowledge of Organic Chemistry is expected of those attending the Class, which should, however, be taken concurrently with the preceding Course, and with Practical Organic Chemistry in the Laboratory.

Fee:—For the Course, £6 6s.; for a Term, £2 12s. 6d.; for a Second Course, £3 3s.

An Advanced Course will be held twice a week during the second and third terms, for those engaged in prosecuting research in Organic Chemistry. Fee, £2 12s. 6d.

Practical Classes.

Practical Classes in Inorganic and Organic Chemistry are conducted by the Assistants.

Analytical and Practical Chemistry.

The Laboratory is open daily from 9 a.m. to 4 p.m., Saturdays excepted, from October until the middle of July, with a short recess at Christmas and at Easter.

Fees: for the Session, £26 5s.; six months, £18 18s.; three months, £10 10s.; one month, £4 4s.

Three specified days a week:—for the Session, £15 15s.; six months, £11 11s.; three months, £6 6s.; one month, £2 12s. 6d., exclusive of expense of materials. Students may enter at any period of the Session.

When accompanied by, or preceded by, attendance on the Lectures on Inorganic and Organic Chemistry, the Laboratory Course qualifies Students in the application of Chemistry to Manufactures, Metallurgy, Medicine, or Agriculture, &c.

There is also a Chemical Library containing the chief Journals and Standard Works on Chemistry.

Certificates of Honour are granted to competent Students on the work done during the Session. Several valuable Scholarships are available to students.

ROYAL COLLEGE OF SCIENCE AND ROYAL SCHOOL OF MINES.

Professor of Chemistry—W. A. Tilden, D.Sc., F.R.S.

Demonstrators—H. Chapman Jones and M. O. Forster, Ph.D., B.Sc.

Assistants—G. S. Newth, J. C. Philip, M.A., Ph.D., B.Sc., and H. Burrows, Ph.D.

The Royal College of Science at South Kensington is intended, primarily, for the instruction of teachers, and of students of the industrial classes selected by competition in the examinations of the Board of Education. The Royal School of Mines is incorporated with the Royal College of Science. Students entering for the Associateship

of the Royal School of Mines obtain their general scientific training in the Royal College of Science. The instruction in the Royal College of Science is arranged in such a manner as to give the Students a thorough training in the general principles of Science, followed by advanced instruction in one or more special branches of Science. The Associateship is granted in certain divisions or lines of study. Students who go through any one of the prescribed courses of instruction and pass the necessary Examinations receive a Certificate of Associateship of the Royal College of Science, or of the Royal School of Mines. Students who are not candidates for the Associateship are permitted to enter as occasional students in one or more special branches of science. The Associateship of the Royal College of Science is given in one or more of the following divisions:—Mechanics, Physics, Chemistry, Biology, and Geology, and the Associateship of the Royal School of Mines in Metallurgy and Mining.

The course of instruction lasts for three years, is the same for all the divisions during the first year, after which it is specialised according to the particular division in which the student is working for the Associateship.

Examinations are held at the end of each course of instruction and at such other periods as may be found necessary. On the results of these examinations the successful candidates are arranged in two classes, first and second. There are also "Honours" examinations for the subjects of the third year, the successful candidates being placed in order of merit. A student obtains the Associateship who passes in all the subjects of the first year, and, in the second and third year, those subjects prescribed as necessary for the division in which he seeks to obtain his Associateship. A student who goes through the prescribed course of instruction in any subject and passes the necessary examinations receives a certificate to that effect.

Students who do not wish to attend the lectures are admitted for short periods to the laboratories, at the discretion of the Professors. The fees for the laboratories are £4 per month.

Students not entering for the Associateship are admitted to any particular course of study, so far as there is room, on payment of the fees shown in the following table:—

	Lectures.	Laboratory.
Chemistry	6	13
Physics	5	12
Biology with Botany .. .	5	12
Geology with Mineralogy ..	4	8
Mechanics	4	6
Metallurgy	2	13
Mining	4	
Astronomical Physics .. .	2	3
Mine Surveying	10	

The fees for the first two years amount to about £75, and for the remainder of the course for the Associateship they vary from £30 to £40.

The Session is divided into two Terms. The first Term begins about the first week of October, and ends about the middle of February. The second Term begins in the middle of February, and ends about the middle of June.

The hours of study are from 10 a.m. to 1.15, and from 2 to 4 p.m. every day, except on Wednesday and Saturday, when the College closes at 1 p.m.; students receiving instruction in Mathematics and Mining attend till 5 p.m. on specified days.

Several valuable Exhibitions, Scholarships, and Prizes are attached to the studentship.

Summer Courses for Teachers.—Short courses of instruction are given annually, about July, in different branches of science for the benefit of teachers of science schools in the country. The courses last three weeks. About 250 teachers are admitted to them, and they receive third class railway fare to and from South Kensington, and a sum not exceeding £3 towards their expenses.

THE SCHOOL OF THE PHARMACEUTICAL SOCIETY OF GREAT BRITAIN.

The Sixty-first Session will commence on Oct. 1, 1902.

Professors—Chemistry, W. Palmer Wynne, D.Sc., F.R.S., F.I.C.; Botany, J. Reynolds Green, Sc.D., F.R.S., F.L.S.; Pharmaceutics, Henry G. Greenish, F.I.C., F.L.S. (Dean).

A Course of Lectures on Physical, Inorganic, and Elementary Organic Chemistry commences in October and terminates at the end of June. An Advanced Course of Lectures begins in October and extends to the end of March. These Lectures are adapted to the requirements of Pharmaceutical and Medical Students, and also those who are proceeding to degrees at the University of London, or who are preparing for the examinations of the Institute of Chemistry.

Entries may be made for single classes. Certificates of attendance at the two Courses of Lectures on Chemistry and at the Chemical Laboratories are accepted as evidence of chemical training by the Institute of Chemistry in connection with the Examinations for the Associateship, and also by the conjoint Board of the Royal Colleges of Physicians and Surgeons, as well as by other examining bodies.

Prospectuses and further information may be obtained from Mr. Richard Bremridge, Secretary and Registrar, 17, Bloomsbury Square, London, W.C.

UNIVERSITY COLLEGE OF WALES, ABERYSTWYTH.

UNIVERSITY OF WALES.

Professor—J. J. Sudborough, D.Sc. (Lond.), Ph.D. (Heidelberg), F.I.C.

Demonstrators—H. Hibbert, M.Sc. (Vic.), and A. Brooke, Ph.D. (Strassburg).

Lecturer in Agricultural Chemistry—J. Alan Murray, B.Sc. (Edin.).

Lecturer on Dyeing—E. J. Wilkinson.

The College is open to male and female students above the age of sixteen years. The Session commences on October 1st, on which day all Students will be expected to meet the Professors in the Examination Hall of the College.

Lecture Courses.—(1) Matriculation Course; two lectures weekly throughout the Session. (2) Intermediate Science Course; four lectures weekly throughout the Session. (3 and 4) B.Sc. Courses; A, three lectures weekly on Organic Chemistry; B, three lectures weekly on General and Physical Chemistry. (Courses A and B will generally be given in alternate Sessions; for 1902-1903, Course A). (5 and 6) Courses in Agricultural Chemistry. For students in their first year, 3 lectures, and for those in their 2nd year, 2 lectures weekly during the Michaelmas and Lent terms.

Laboratory Courses.—The Laboratories are open daily from 10 a.m. to 1 p.m., and from 2.15 to 5 p.m., except on Saturdays. Classes for the Systematic Study of Qualitative and Quantitative Analysis will be formed, and Special Courses will be arranged for those who intend to follow Medicine or Pharmacy, or any one particular branch of Applied Chemistry, always provided that such Students possess the requisite knowledge of Theoretical Chemistry. The hours will be arranged, as far as possible, to suit the requirements of the individual Student.

The College is recognised by the University of Edinburgh and the Royal University of Ireland, and by the Colleges of Physicians and Surgeons of England, Scotland, and Ireland as an institution at which the instruction necessary for their respective Diplomas in Medicine, in Chemistry, Physics, and Biology may be given. One year for graduation in Medicine and two years for graduation in Science may be spent at Aberystwyth.

Fees.—The Fee for the whole Session, if paid in advance, is £10; if paid by Single Terms, for the first term of attendance in each Session, £4; for the second term, £3 10s.; for the third term, £3. These composition fees enable the Student to attend any or all the Classes of the

College, with the exception that a small extra fee is charged for Laboratory Instruction. Thus, for Practical Chemistry, the additional fee is, for six hours' work per week, 12s. 6d. per term, and for twelve hours, 25s. per term. The fees for those who desire to spend several days weekly in the laboratory may be learned on application to the Registrar. Fee for a single Lecture Course £1 per term.

Scholarships and Exhibitions varying in value from £10 to £40 per annum will be offered for competition at examinations which commence on September 18, and exhibitions are awarded at the end of the Session on the results of the class examinations.

Intending Students requiring further information are recommended to write to the Registrar for a copy either of the General Prospectus or of one of the Special Prospectuses issued for the Agricultural and Normal Departments.

UNIVERSITY COLLEGE OF NORTH WALES, BANGOR.

A CONSTITUENT COLLEGE OF THE UNIVERSITY OF WALES.

Chemistry.—Professor, James J. Dobbie, M.A., D.Sc. Demonstrator, Alexander Lauder, B.Sc., F.C.S. Assistant Lecturer in Agricultural Chemistry, Alan Baguley, B.Sc. Scholar Assistant, C. K. Tinkler.

Physics.—Professor, E. Taylor Jones, D.Sc.

The Session opens October 1st, 1902. All regular classes are open to men and women students above the age of 16 years. The following Courses of Lectures will be given.

Matriculation Course.—Subjects: Those prescribed for the Matriculation Examination of the University of Wales. Fee for the Session £3 3s.

Intermediate Course.—Inorganic Chemistry and Elementary Physical Chemistry. Fee for the Session, £3 13s. 6d.

B.Sc. Course.—Organic Chemistry. Fee for the Session, £3 3s.

Agricultural Chemistry.—Fee, £2 2s.

Laboratory Courses.—The laboratory is open on five days of the week from 10 a.m. to 4 p.m. for instruction in Chemical Analysis and in the Application of Chemistry to Medicine and the Industrial Arts. Fees: six hours per week, £1 1s. per Term; twelve hours, £2 2s.; eighteen hours, £3 3s.; twenty-four hours, £4 4s. Composition Fee for all Laboratory Classes of the Intermediate Science Course taken in one year, £4 4s.

The Chemistry, Botany, Zoology, and Physics Courses are recognised for Medical graduation in the Universities of Edinburgh and Glasgow, and students can make one *Annus medicus* at the college. The Science Courses are recognised for part of the science degree course of the University of Edinburgh.

UNIVERSITY COLLEGE OF SOUTH WALES AND MONMOUTHSHIRE, CARDIFF.

Professor—C. M. Thompson, M.A., D.Sc., F.C.S.

Demonstrators—E. P. Perman, D.Sc., F.C.S., and A. A. Read, F.I.C., F.C.S.

The Session commences October 7th, and terminates on June 20th, and is divided into three terms.

The Junior Course (delivered during the Michaelmas term only) consists of about 50 lectures, and will cover the subjects prescribed for the Matriculation examinations of the University of Wales and the University of London Fee, £2 2s. A revision class is held in the Summer term.

The Intermediate Course consists of about 80 lectures held during the Lent and Summer terms in continuation of the Junior Course, and is the qualifying course for the Intermediate Examination of the University of Wales. Together with laboratory practice, it will cover the subjects required for the Intermediate Examination in Science and the Prel. Sci. (M.B.) Examination of the University of London. Fee, £4 4s.

The Senior Course consists of about 80 lectures on Inorganic Chemistry; Fee, £3 3s.

A course of 20 lectures on Qualitative Analysis and a short course on Organic Chemistry will also be given.

The following lectures on Metallurgy will be given by Mr. Read:—10 lectures on Fuel; Fee, 10s. 6d. 20 lectures on General Metallurgy; Fee, £1 1s. 30 lectures on the Manufacture of Iron and Steel; Fee, £1 1s. A practical course on Iron and Steel Analysis will also be held, and practical instruction in Dry Assaying will be given in the Metallurgical Laboratory, which is fitted with the necessary furnaces and apparatus.

In the laboratory each student works independently, so that the course of study may be adapted to the requirements of the individual. Hours, 9 to 1 and 2 to 5; Saturday, 9 to 1. Fees—Six hours per week, £3 3s. per session; twelve hours, £2 2s. per term; eighteen hours, £3 3s. per term; twenty-four hours, £4 4s. per term.

Registered medical students can prepare for the Intermediate M.B. Examination of the University of London, and spend three out of their five years of medical study in Cardiff. Medical students wishing to graduate at a Scottish University, or preparing for a Conjoint Board Surgical and Medical Diploma, or for the Diploma of the Society of Apothecaries, can spend two years in Cardiff. For further information see the prospectus of the Faculty of Medicine, which may be obtained from the Registrar.

The College is recognised as an institution at which two years of the course for the degree of Bachelor of Science of the University of Edinburgh may be spent.

Students by making a payment of £10 at the commencement of each session may compound for all lecture fees for the whole session. Laboratory fees are not included in the composition fee, but Students preparing for the Science Examinations of the University of Wales and of the University of London may, by making a payment of £13 13s. at the commencement of each Session, compound for both Lecture and Laboratory Fees during the Session.

At the entrance examination in September, and the annual examination in June, several scholarships and exhibitions are awarded. Great importance is attached to special excellence in one subject.

The College Prospectus, and also further information as to scholarships, may be obtained from the Registrar.

A Hall of Residence for Women Students is attached to the College.

UNIVERSITY COLLEGE, BRISTOL.

Professor of Chemistry—Sydney Young, D.Sc., F.R.S.

Lecturer—Francis E. Francis, M.Sc., Ph.D.

Demonstrator—Oliver C. M. Davis.

The session 1902-1903 will begin on October 7. Lectures and classes are held every day and evening throughout the Session. In the Chemical Department lectures and classes are given in all branches of theoretical chemistry, and instruction in practical chemistry is given daily in the chemical laboratory. The department of experimental physics includes various courses of lectures arranged progressively, and practical instruction is given in the physical and electrical laboratories. The Department of Engineering and the Constructive Professions is designed to afford a thorough scientific education to students intending to become engineers, or to enter any of the allied professions, and to supplement the ordinary professional training by systematic technical teaching. This department includes courses specially arranged for students intending to become civil, mechanical, electrical, or mining engineers, surveyors, or architects. Those who attend the mechanical engineering course enter engineering works during the six summer months, and, in accordance with this scheme, various manufacturing engineers in the neighbourhood have consented to receive students of the College into their offices and workshops as articled pupils at reduced terms. Medical education is provided by the Faculty of

Medicine of the College. Several Scholarships are tenable at the College. Full information may be obtained from the Registrar and Secretary.

DAY LECTURES. *Inorganic Chemistry.*

The Courses treat of the principles of Chemistry, and of the Chemistry of the Non-Metals and Metals.

Elementary Course.—Two Lectures a week will be given during the Session. Fee, £4 4s.

Special Course.—A special course of Lectures is also given to Engineering Students.

Intermediate Course.—Three Lectures a week will be given throughout the Session. Fee, £5 5s. There will be tutorial classes in connection with the Elementary and Intermediate Courses.

Advanced Course (Parts I. and II.).—One Lecture a week in each part will be given throughout the Session. Fee for each course, £2 12s. 6d.

Organic Chemistry.

This Course will relate to the more important groups of the Compounds of Carbon.

Two Lectures a week will be given throughout the Session. Fee, £3 3s. An advanced course of lectures will also be given one day a week during the session. Fee, £2 12s. 6d.

Practical Chemistry.—Laboratory Instruction.

The Laboratory will be open daily from 10 a.m. to 5 p.m., except on Saturdays, when it will be closed. Instruction will be given in the Laboratory in all branches of Practical Chemistry, including Qualitative and Quantitative Inorganic and Organic Analysis, the preparation of Chemical Products, Metallurgy, and Inorganic and Organic Research. Special facilities will be afforded to those who desire to study Practical Chemistry as applied to the different processes employed in the Arts and Manufactures. Fees in Guineas—

	5 Days a Week.	4 Days a Week.	3 Days a Week.	2 Days a Week.	1 Day a Week.
Per Session ..	15	12½	10	7½	5
„ Two Terms ..	11	9	7½	5½	3½
„ One Term ..	7	6	4½	3½	2½

Students may arrange to divide their days of laboratory work into half-days.

Chemical Scholarship.—Among others, a Chemical and a Metallurgical Scholarship each of £25 are offered for competition.

EVENING LECTURES.

Two courses of Lectures will be delivered during the First and Second Terms; they will be devoted to the consideration of the general Principles of Chemistry and Chemical Physics and the Chemistry of Non-Metallic and Metallic Elements. Special attention will be paid throughout to those products which have a practical application in the Arts and Manufactures. Fee for each course, 7s. 6d.

Pharmaceutics.—During the second and third Terms a course of Lectures will be given one evening a week dealing chiefly with the Materia Medica, Pharmacy, and Chemistry of the British Pharmacopoeia. Fee, £1 1s.

Practical Chemistry.—Laboratory Instruction.—The Laboratory will be open two evenings a week from 7 till 9. Instruction will be given in Qualitative and Quantitative Analysis, and in the Preparation of Chemical Products. Fees:—(Two Terms) Two Evenings, 25s.; One Evening, 15s. (One Term) Two Evenings, 15s.; One Evening, 10s. 6d.

University College, Bristol, has been approved by the Council of the Institute of Chemistry as a College at which all the subjects required for the admission of Associates to the Institute are taught.

The Calendar of the College, price 1s. (post-free, 1s. 4d.), containing detailed information of the various Courses, may be obtained on application to the Registrar and Secretary.

MERCHANT VENTURERS' TECHNICAL COLLEGE, BRISTOL.

CHEMISTRY AND METALLURGY.

Professor—J. Wertheimer, B.Sc., B.A., F.I.C., F.C.S.

Lecturers—G. P. Darnell-Smith, B.Sc., F.I.C., F.C.S.;

H. A. M. Borland, A.R.C.S.; A. J. Carrier, B.Sc.;

Demonstrators—F. B. Gatehouse; H. C. Shilstone;

H. R. Tutton.

Assistant in Chemical Laboratory—C. E. Young.

The Laboratories of this Department are open during the Term on every week-day as a School of Practical Chemistry, Analysis, and Assaying, to Students of either sex. They include the Junior Laboratory (for forty Students working at one time), Senior Laboratory, Gas Analysis Room, Balance Room, Combustion Room, Store Rooms, and Metallurgical Laboratory. Each Student works independently, and receives individual instruction and help.

The training in this Department is intended to fit Students to become Professional Chemists, to prepare them for Medical or Pharmaceutical examinations in Chemistry, to instruct them in the branches of Applied Chemistry required in Manufactures and Agriculture, and to train them in practical Metallurgical processes, especially Assaying.

Special facilities will be given to Students wishing to undertake research work approved by the Principal; and the Courses provide completely for the Preliminary Scientific Examination (M.B.), and for the Examinations in Chemistry for the B.Sc. degree of the University of London.

The three years' course at this College in the subjects of Theoretical and Practical Chemistry, Theoretical and Practical Physics, and Elementary Mathematics, is accepted by the Council of the Institute of Chemistry as satisfactory evidence of the training of candidates for the Associateship of the Institute, provided the details in each case be certified by the Professors.

Certificates of Proficiency in Assaying will be given to Students who complete a Course of not less than two years' work in the Metallurgical Department, and pass satisfactorily the College examinations in the Theory and Practice of Assaying.

The Laboratories are open every week-day from 9 till 12 noon, and from 1.30 to 4.30 p.m., except Saturday, when they close at noon.

The Annual Fee of £10 10s. secures (in addition to the use of either or both Laboratories) permission to attend the Lectures in Physics and Mathematics as well as Chemistry, and to use the Physical Laboratory and the Engineering Workshop.

The College Evening Classes, available to persons of either sex, will commence on Monday, September 29th.

Further detailed information may be obtained from the College Prospectus, price 6d.

UNIVERSITY OF BIRMINGHAM.

Professor—Percy F. Frankland, Ph.D., M.Sc., LL.D., F.R.S.

Assistant Lecturers and Demonstrators—T. S. Price, Ph.D., D.Sc.; Alex. Findlay, M.A., D.Sc., Ph.D.; Alex. McKenzie, M.A., D.Sc., Ph.D.

The Session will be opened on October 1st, 1902.

Lecture Courses.

First Year.—A. This part of the course is arranged (1) to give a full exposition of the general principles of Chemical Science, (2) for the systematic study of the properties of the more important elements and their compounds, and (3) to indicate the chief applications of Chemistry in the Arts and Manufactures. Four hours weekly during the Winter and Spring terms. At least one of the above meetings of the class in each week will be devoted to tutorial work. Attendance at this tutorial class is compulsory, as is the performance of the weekly exercises set by the Professor. Mondays to Fridays inclusive, at 9.30 to 10.30 a.m. Fee, £5 5s. B. This part of the course in-

cludes an introduction to the study of Organic Chemistry, with a description of the properties, relations, and methods of preparation of the more important groups of Carbon Compounds. Three hours weekly during the Summer term. Mondays, Wednesdays, and Fridays, at 9.30 to 10.30 a.m. Fee, £1 11s. 6d.

Second Year.—Advanced Organic Chemistry.—This course extends over two years, and is divided into two parts:—(a) Carbon Compounds of the Fatty Series; (b) Aromatic and other Cyclic Compounds. Only one of these parts will be taken in each year. The class meets twice weekly by arrangement during the Winter and Spring terms. Fee, £2 2s. **General and Physical Chemistry.**—This course will deal in outline with various Chemical subjects. The class meets once weekly by arrangement during the three terms. Fee, £1 11s. 6d.

Third Year.—A further Course in Advanced Organic Chemistry will deal with one of the above parts of the Course. The class meets two hours weekly by arrangement during the Winter and Spring terms. Fee, £2 2s. A further Course on General, Physical, and Organic Chemistry, in which special subjects attracting attention at the time receive treatment. Fee, £1 11s. 6d.

Practical Chemistry.

The instruction in Practical Chemistry extends over three years. The Laboratory will be open daily from 9.30 to 5, except on Saturdays, when it will be closed at 1 p.m. Fees—

	All day.	Three hours per day.	Three hours per day; five days a week.	Three hours per day; three days a week.
	Guineas.	Guineas.	Guineas.	Guineas.
One Term ..	7	4½	4	2½
Two Terms ..	13	8½	7½	5
Three Terms ..	18	12	11	6½

A Course of short demonstrations and exercises is given by the Professor or one of his Assistants once a week. All first-year Students are required to attend, unless exempted for special reasons by the Professor. No Fee.

Special facilities are given to Advanced Students for the prosecution of original research.

Metallurgy.

There is a separate laboratory for metallurgical students in which provision is made for instruction in assaying, &c.

Scholarships.

Priestley Scholarships.—Three Open Scholarships in Chemistry of the value of about £96 each are awarded annually in September.

Bowen Scholarship.—One Open Scholarship in Metallurgy of the value of about £96 is awarded annually in September.

For particulars apply to the Registrar.

Excursions.

During previous Sessions permission has been obtained to visit some of the great factories in or near Birmingham, in which chemical and metallurgical industries are carried on. Students have thus had most valuable opportunities of gaining a practical acquaintance with some branches of Applied Science. The privilege thus courteously granted by several manufacturers will, it is hoped, be enjoyed in every future Session. The excursions will be conducted by the Professor or Lecturers.

BRADFORD MUNICIPAL TECHNICAL COLLEGE.

DEPARTMENT OF CHEMISTRY AND DYEING.

Head of Department.—W. M. Gardner.

Lecturers in Chemistry.—B. North, A.R.C.Sc. (Lond.); S. F. Stoll, F.C.S.; and W. Trotter.

Lecturer in Dyeing.—A. B. Knaggs, F.C.S.

Lecturer in Metallurgy.—W. J. Willis.

Lecturer in Gas Manufacture.—W. Cranfield.

Lecturer on Botany, Biology, and Pharmacy.—W. West, F.L.S., Ex. Pres. Yorkshire Naturalists' Union.

The following courses of instruction are provided:—

I. General Chemistry Course, extending over three years, including Lectures in Inorganic, Organic, and Technological Chemistry, Principles of Analysis, Technical Analysis, Chemical Philosophy, Crystallography, Fuels, Lighting and Ventilation, Physics, Mathematics, Mechanics, with Laboratory work in Chemistry, Physics, Bacteriology, and Microscopy.

II. Chemistry and Dyeing, extending over three years. Includes most of the above subjects, along with Lectures and practical work in Dyeing, Colour matching, &c.

III. Chemical Engineering. Three years' course, preparing Students for positions in Chemical Works, Sewage Works, &c.

IV. Sanitary Science. One year's Course, recognised by the Sanitary Institute as preparing for their certificate examination. Subjects: Chemistry, Physics, Sanitary Engineering, Sanitary Law, Building Construction, Drawing, Physiology, and Bacteriology.

V. Dyeing. Extending over one or two years.

VI. Textile and Dyeing. Arranged for those Students who desire to study the two subjects simultaneously.

VII. General Pharmaceutical Course. Prepares for the Minor and Major Pharmaceutical Examinations. Each extends over two years on three half-days per week, and includes Chemistry and Physics, Botany, Biology, Materia Medica, Pharmacy, and Dispensing.

VIII. Attendance at the College Courses and College Certificates in Chemistry, Physics, Animal Biology, and Botany are recognised by the Conjoint Board for Medical Studies.

ROYAL AGRICULTURAL COLLEGE, CIRENCESTER.

CHEMICAL DEPARTMENT.

Professor.—Prof. E. Kinch, F.C.S., F.I.C.

Assistant.—W. James.

Systematic courses of Lectures are given on the various branches of Chemistry in its relation to Agriculture, illustrated by experiments, and by the collections in the College Museum. They comprise the laws of Chemical Combination and the general Chemistry of mineral bodies, and of the more frequently occurring bodies of organic origin, with the relationships of their leading groups; and, finally, the applications to practical operations of the Chemistry of the atmosphere, of soils and manures, of vegetation, of stock feeding, and of the processes and products of the dairy.

In the Laboratory practical instruction is given in the construction and use of apparatus and in Chemical manipulation and analysis, both qualitative and quantitative. After studying the simple operations and the properties of the commonly occurring substances, the Students are taught to analyse a series of compounds, and apply the knowledge thus obtained to the analysis of manures, soils, waters, feeding stuffs, dairy products, and other substances met with in the ordinary course of Agricultural practice. Chemico-agricultural researches are undertaken by the senior Students under the direction of the Professor and his Assistants.

VICTORIA UNIVERSITY.

THE YORKSHIRE COLLEGE, LEEDS.

Professor of Chemistry.—Arthur Smithells, B.Sc., F.R.S.

Lecturer in Organic Chemistry.—Julius B. Cohen, Ph.D.

Assistant Lecturers and Demonstrators.—T. S. Patterson,

Ph.D.; J. McCrae, Ph.D., and H. M. Dawson, B.Sc., Ph.D.

Demonstrator.—C. E. Whiteley, B.Sc.

The Session begins October, 1902.

Lecture Courses.

1. General Course of Chemistry.—Monday, Wednesday, and Friday, at 11.30 a.m. Fee for the Course, £4 4s.

2. Advanced Inorganic Chemistry.—Metals. Monday, Wednesday, and Friday, at 9.30 a.m. Fee, £3 13s. 6d.

3. Advanced Inorganic Chemistry.—Non-metals. Tuesday, Thursday, and Saturday, at 9.30 a.m. Fee, £3 13s. 6d.

4. Organic Chemistry.—Tuesday, Thursday, and Saturday at 12 noon Fee £3 13s. 6d.

5. Honours Courses—

- (a) Organic Chemistry.—Wednesday and Friday at 12 noon. Fee, £2 12s. 6d.
 (b) History of Chemistry.—Monday and Wednesday at 9.30 a.m. in the First Term. Fee, £1 1s.
 (c) Physical Chemistry.—Thursday and Saturday at 9.30 a.m. in the Second and Third Terms. Fee, £2 2s.
 (d) Selected subjects.—Tuesday at 9.30 a.m. in the Second and Third Terms. Fee, £1 1s.
 (e) Electro-chemistry.—One hour weekly. 31s. 6d.

Laboratory Courses.

The College Laboratory will be open daily from 9 a.m. to 1 p.m., and from 2 to 5 p.m., except on Saturdays, when it will close at 1 p.m.

Fees for the Session—Students working six days per week, £21; five, £18 18s.; four, £16 16s.; three, £13 13s.
Practical Course in Sanitary Chemistry.—Tuesdays and Thursdays from 2 to 5 p.m., from January to March. Fee, £5 5s.

Elementary Science for Teachers.—Saturdays, 9.30 to 12.30, for half the session. £2 12s. 6d.

Dyeing Department.

Professor—J. J. Hummel, F.I.C.

Lecturer and Research Assistant—A. G. Perkin, F.R.S.E.

Assistant Lecturer—A. B. Steven, B.Sc.

This Course extends over a period of three years, and is intended for those who wish to obtain a full scientific and practical education in the art of dyeing. It is suitable for those who purpose in the future to take any part in the direction of the operations of dyeing or printing of textile fabrics, e.g., the sons of manufacturers, calico printers, managers, master dyers, &c.

Leather Industries Department.

Professor—H. R. Procter, F.I.C.

Assistant Lecturer and Demonstrator—Andrew Turnbull, Ph.D.

Demonstrator—F. Austyn Blockey.

The full Courses, which extend over a period of either two or three years, are suitable to all who intend to become Technical Chemists in the Leather Industry, or managers of important works, and are recommended to sons of tanners. The Courses include instruction in chemistry, a modern language, leather manufacture, and practical work in the Leather Industries Laboratory and Dye-house.

Agricultural Department.

Professor—R. S. Seton, B.Sc.

Lecturer in Agricultural Chemistry—H. Ingle, F.I.C.

The full Course occupies two years, and includes instruction in chemistry, physics, mathematics, geology, botany, forestry, engineering and surveying, and the principles of agriculture, as well as practical work in the various laboratories and out-door agriculture.

Research Students are admitted to the College Laboratories on reduced terms.

Several valuable Scholarships are at the disposal of the College, viz., the Salt, Akroyd, Brown, Baines, Emsley, Craven, Leeds City Council, and Clothworkers' Scholarships, and one of the 1851 Exhibition Scholarships. The North, East, and West Ridings County Council's Scholarships are tenable at the Yorkshire College.

UNIVERSITY COLLEGE, LIVERPOOL.

Professor—J. Campbell Brown, D.Sc.

Lecturer on Organic Chemistry—A. W. Titherley, D.Sc., Ph.D.

Lecturer on Metallurgy—T. L. Bailey, Ph.D.

Demonstrators and Assistant Lecturers—T. L. Bailey, Ph.D.; A. T. de Moulpiéd, B.Sc., Ph.D.; and A. W. Titherley, D.Sc., Ph.D.

Assistant—H. H. Froyssell.

The Session commences October 2nd.

Entrance Scholarship Examination takes place early in May each year.

The Classes meet the requirements of candidates for the Ordinary B.Sc. Degree, for Chemistry Honours, or for the M.Sc. or D.Sc. Degree in Victoria University; for Degrees in Medicine of Victoria, London, and Edinburgh; for the Pharmaceutical Diplomas; for a special Technological Certificate of University College; and for those studying Chemistry as a preparation for professional, technical, or commercial life. The Classes qualify for the Fellowship of the Institute of Chemistry of Great Britain and Ireland, and other Examination Boards.

Lecture Courses.

General Elementary Course on the principal non-metallic elements and the most important metals, the principles of Chemical Philosophy, and an introductory sketch of Organic Chemistry. Three Terms. Fee, £4.

Engineer's Course of Lectures with Practical Class.

Two Terms. Fee, including Practical class, £4.

Pharmacy Courses: Junior, £3; Senior, £3.

Dental Course, Lectures and Practical. Fee, £5 5s.

Course A.—Non-metals. Fee, £3.

Course B.—Metals. Fee, £3.

Course C.—Organic Chemistry. Fee, £3.

Course H.—Special Organic Subjects. Fee, £2.

Course D.—Physical Chemistry, with Practical Work. Fee, £2.

Course E.—History of Chemistry and of the Development of Modern Chemical Philosophy. Three Terms. Fee, £2.

Courses F.—Applied Chemistry and Metallurgy: Lectures on Technology are given in connection with Laboratory work at hours to be arranged. The subjects are varied in different years. (1) Alkali and Allied Manufactures. (2) General Principles of Metallurgy. (3) Iron, Steel, and Aluminium. (4) Copper, Lead, Silver, and Gold, and other Metals. (5) Distillation of Coal and Tar Industries. (6) Fuel and Gas. (7) Chemistry Applied to Sanitation. (8) Technical Gas Analysis. (9) Electro-chemical work. Three terms. Fee, each course £1 10s.

Practical Classes.

(1) Preliminary. (2) Intermediate: Qualitative Analysis of Inorganic Substances and of some of the more common Organic Substances; preparation of Inorganic Compounds. (3) Revision Class. (4) Senior: Practical Organic. (5) Practical Exercises on Technology, Pharmaceutical Chemistry, Sanitary subjects, Examination of Water and Air, &c. (6) Quantitative Class.

Chemical Laboratory.

The Chemical Laboratories recently erected provide accommodation for every kind of chemical and metallurgical work and research. They include thirty rooms devoted to the study of Chemistry and Metallurgy. Separate rooms are provided for Organic Work, Quantitative Analysis, Water Analysis, Gas Analysis, Photometry and Photography, Electro-chemical Work, Metallurgy Laboratories, and a Research Laboratory. New stores for students' apparatus and chemicals have also been built and placed in charge of a skilled dealer.

Students desirous of gaining a thorough theoretical and practical acquaintance with Technical Chemistry, or who intend to adopt Chemical work as a profession, must devote three or four years to special study, for which a full curriculum is provided.

TABLE OF FEES.

	One Term, Three Months.	Three Terms, One Session.
Per Week.		
One day	£4	£7
Two days	5 10s.	10
Three days	7	13
Whole week	10 10s.	21

Pharmaceutical Course (see special syllabus).

Technological Curriculum.

Preliminary Year.—Chemistry, the Elementary Course. Practical Classes 1 and 2. Mathematics, or Mechanics,

Elementary Engineering, Drawing, and Design (in this or one of the following years). German. Or, the Victoria Preliminary Course and Examination may be taken.

First Year.—Chemistry—Courses A and B; Chemical Laboratory three days per week; Technological Chemistry, Course F. Physics, with laboratory work, one day per week. Mathematics (intermediate). German. Engineering, First Year Course, Autumn and Lent Terms. Intermediate B.Sc. Examination may be passed.

Second Year.—Chemistry, Lecture Course C, on Organic Chemistry, Lecture Course D or E, Technological Chemistry, Course F. Chemical Laboratory, four days per week. Engineering, Mathematics, or Physics (Advanced). The Final Examination for the Victoria B.Sc., or the Intermediate Examination of the Institute of Chemistry, may be taken.

Third Year.—In the third year the student should begin to specialise. The following are possible alternatives:—1. Courses D, E, and F; Course H. Any other Courses omitted in a previous year. Laboratory, five days per week. The Degree of B.Sc. in the Honours School of Chemistry may be taken. 2. Metallurgical Classes. Metallurgical Laboratory, two days per week. Chemical Laboratory, Gas Analysis, and Electro-chemistry, two days per week. Physical and Electrotechnical Laboratory, one day per week. 3. Courses D, E, and F. Physics (Senior Course). Chemical Laboratory, four days per week. Physical Laboratory, one day per week. 4. Courses D and F, and a course on Chemistry applied to manufacturing operations. Chemical Laboratory, three or four days per week. Engineering Laboratory and Electrotechnical Laboratory, one day per week.

Students may finally choose a special subject either of research or of applied Chemistry. The Final Examination for the Associateship of the Institute of Chemistry may be taken after 1, 2, or 3. Those who have taken the Ordinary Degree of B.Sc. may pass the Victoria M.Sc. Examination in any subsequent year.

The Sheridan Muspratt Chemical Scholarship of £50 per annum, tenable for two years, will be competed for in December, 1902, on an Examination in subjects which are included in the first two and a half years of the above curriculum. Candidates should send in their names to the College Secretary not later than November 15, 1902. Other Scholarships, Entrance Scholarships, and Free Studentships are also available to Students.

Evening Classes.

Classes, including laboratory work, will be held during the winter.

The Prospectus containing full particulars may be obtained from the Secretary, University College, Liverpool.

DURHAM COLLEGE OF SCIENCE, NEWCASTLE-ON-TYNE.

Professor of Chemistry—P. Phillips Bedson, M.A., D.Sc., F.I.C., F.C.S.

Lecturer in Chemistry—F. C. Garrett, M.Sc., F.C.S.

Lecturer in Agricultural Chemistry—S. Hoare Collins, F.I.C., F.C.S.

Assistant Lecturers and Demonstrators—J. A. Smythe, M.Sc., Ph.D., and H. W. Cousins, B.Sc.

First Year Courses.—1. *General Course.*—This Course of Lectures will extend over the three terms of the Session, and is intended to serve as an introduction to the Science. The Lectures will be of an elementary character, and whilst framed to meet the requirements of First Year Students will also be serviceable to such as intend pursuing Chemistry in its various applications in the arts and manufactures, as, for instance, Brewing, Metallurgy, the Manufacture of Soda, Soap, Glass, &c. The subjects treated will include an exposition of the Principles of Chemistry, and a description of the preparation and properties of the chief Elementary Substances, both metallic and non-metallic, and their more important native and artificial compounds. The class will meet on Mondays, Wednesdays, and Fridays, at 11 a.m., and will

commence on Wednesday, October 8th. Fee, £3 10s. for the Session.

11. *Special Course.*—More advanced than the above. Mondays and Fridays, 11 to 12. Fee, £3 10s. for Session.

Second Year Courses.—The Lectures for the second year students consist of a course of Lectures on Organic Chemistry, and a course of Lectures on Inorganic Chemistry. The class on Organic Chemistry will meet on Tuesdays and Thursdays at 11 a.m., and will commence on October 7th. The class for Inorganic Chemistry meets at 11 a.m. on Mondays. Fee for Session, £3 10s.; for Inorganic alone £1 10s.; and for Organic alone £3.

Third Year Courses.—Advanced Classes are held for the study of Inorganic, Organic, and Theoretical Chemistry. Fee for the course, £3 10s. Also special class in Crystallography.

A Lecture Course in Analytical Chemistry will be given on Fridays, at 9.15 a.m.

Metallurgy and Assaying.—Lecturer, Professor Louis, M.A., F.I.C., F.C.S.; Demonstrator, G. H. Stanley, A.R.C.M. A Metallurgical Laboratory is provided, in which instruction is given in the ordinary processes of Dry Assaying, and in the preparation and analysis of Alloys, &c. Fees as for Chemical Laboratory.

Agricultural Chemistry.—The instruction in this branch of Chemistry will consist of a series of Lectures and of special practical work in the Chemical Laboratory. Students will be expected to have a knowledge of Elementary Chemistry, such as may be obtained by attending the General Course.

The Lecture Course in Agricultural Chemistry is arranged for two days a week throughout the Session. Fee, £3 10s.

Practical Chemistry.—The Laboratory is open from 10 a.m. to 1 p.m., and from 2 to 5 p.m., except on Saturdays, when it closes at 1 p.m. **Laboratory Fees.**—Students working two days, £2 10s. per term, £6 per session; one day per week, £1 10s. per term, £3 10s. per session.

Courses of Study.—Students will be divided into two classes:—(1) Regular, or Matriculated Students, who are also Members of the University of Durham; and (2) Non-Matriculated Students. Regular Students will be required to follow such a course of study in the subjects professed in the College as will enable them to pass the Examinations for the title of Associate in Science of the University of Durham. Non-Matriculated Students will attend such classes as they may select. Every candidate for admission as a matriculated student must pass an examination on entrance, in reading, writing from dictation, English or Latin Grammar, arithmetic (including decimals), and geography. Registered students in medicine are exempted from this examination, or students who produce a certificate of having passed either of the three following examinations:—

1. The first examination for the Degree of B.Litt.
2. Durham Examination for certificate of proficiency in General Education, held in March and September.
3. Durham Examination for Students in Arts in their first year, or any examination of a similar nature that may be accepted by the Council.

Associateship in Science.—Every candidate for the Associateship in Science will be required to satisfy the examiners in—Mathematics, Physics, Chemistry, and either Geology or Natural History—in an examination to be held at the end of the candidate's first year, and at the end of the second year in an examination in a more advanced stage in any two of these subjects. Associates in Science are admissible one year after obtaining the title of Associate to the examination for the degree of Bachelor of Science of the University of Durham.

Exhibitions.—Three Exhibitions of the value of £25, £15, and £10 respectively will be awarded in October next to Candidates desirous of attending the first year course of study in the College.

The examination will be held at the College, and will commence on Monday, September 29th.

Evening Lectures.—Courses of Evening Lectures will be given, with a Practical Class for Laboratory instruction. Several valuable Scholarships are available for students, including the Johnston Chemical Scholarship of the value of £60 for one year, which is open to Bachelors of Science of any British University.

OWENS COLLEGE, VICTORIA UNIVERSITY, MANCHESTER.

Professor and Director of the Chemical Laboratory—Harold B. Dixon, M.A., F.R.S.

Professor of Organic Chemistry—W. H. Perkin, Ph.D., F.R.S.

Demonstrators and Assistant Lecturers—George H. Bailey, D.Sc., Ph.D.; W. A. Bone, D.Sc.; P. J. Hartog, B.Sc.; D. L. Chapman, B.A.; Norman Smith, B.Sc.; and F. V. Darbishire, B.A., Ph.D.

Demonstrator in Organic Chemistry—F. A. Lidbury, B.Sc.

Lecturer in Technical Organic Chemistry—Jocelyn F. Thorpe, Ph.D.

Professor and Director of the Physical Laboratories—Arthur Schuster, Ph.D., F.R.S.

Assistant Lecturer in Metallurgy—Dr. Bone.

The Session begins on October 7, 1902.

The instruction is given by means of Experimental Lectures and Tutorial Classes. The Chemical Classes form part of the Courses for Chemistry in the University.

Chemistry Lecture Courses.

General Chemistry Course.—Tuesdays, Thursdays, and Saturdays, at 9.30, during the two Winter Terms.

Introduction to Organic Chemistry.—Wednesdays and Fridays, at 9.30, during Lent Term.

These courses are intended for Medical Students and others beginning the study of chemistry.

First Year Honours Course.—Mondays, Wednesdays, and Fridays, 11.30 a.m., during the two Winter Terms. The Non-Metals.

Second Year Honours Course.—Tuesdays, Thursdays, Saturdays, 11.30 a.m., during the two Winter Terms. The Metals.

Third Year Honours Course.—At times to be arranged. Physical Chemistry.

Organic Chemistry (General).—Mondays and Fridays, 9.30, during two Winter Terms.

Organic Chemistry (Advanced).—Tuesdays and Thursdays, 9.30, during the two Winter Terms.

History of Chemistry and Chemical Philosophy.—Wednesdays, 9.30, during the Session.

METALLURGY.—**Lectures:** The Metallurgy of Copper, Lead, Silver, Gold, and the Metallurgy of Iron and Steel will be given in alternate years. **Practical:** The Metallurgical Laboratory will be open to students every day.

ELECTRO-CHEMISTRY.—In connection with the new John Hopkinson Memorial Laboratory, rooms have been fitted for the study of Electricity in its applications to Chemistry. Courses of lectures and laboratory instruction will be given to Advanced Chemical Students in the coming Session. **Lecturer**—R. S. Hutton, B.Sc.

The Chemical Laboratories are open daily from 9.30 a.m. to 4.30 p.m., except on Saturdays, when they are closed at 12.30 p.m.

Courses for B.Sc. Degree.—To qualify for the B.Sc. Degree of the Victoria University, Students have to attend a prescribed course of study extending over three years, and to pass the Preliminary Examination of the University either on entering or at the end of a year's Course.

The Honours Course of Chemistry is as follows:—**First year:** First year Honours Lectures; Mathematics (3 hours a week); Physics (3 hours a week); a Language (3 hours a week); Chemical Laboratory (3 days per week). **Second year:** Second year Honours Lectures; General Organic Lectures; Applied Chemistry Lectures; Physics Laboratory (1 day per week); Chemical Laboratory (3 days per week). **Third year:** Third year Honours

Lectures; Honours Organic Lectures; History of Chemistry Lectures; Chemical Laboratory (5 days per week).

The following awards are made to successful Students in the Honours Examination:—A University Scholarship of £50; a Mercer Scholarship of £25. A University Fellowship of £100 is awarded annually among the Graduates in Science for the encouragement of Research. Among the College Scholarships open to Chemical Students are the Dalton Chemical Scholarship, £50 per annum for two years; the 1851 Exhibition Scholarship; &c.

Applied Chemistry.

First Course.—Sulphuric Acid and Alkali Manufactures. General Principles of Chemical Engineering.

Second Course.—The Chemistry of Fuel. The Manufacture of Illuminating Gas and Gaseous Fuel.

Third Course.—Natural and Artificial Dye-stuffs, and the Principles of Dyeing and Printing.

Certificates in Applied Chemistry.

The course extends over a period of three years, and comprises systematic instruction by means of lectures and practical work in the laboratories.

Before admission to the first year's course students are required to give such evidence of elementary knowledge of Mathematics and Chemistry as shall be considered satisfactory by the Senate.

The first year's course is the same for all students working for the certificate.

In the second and third years a choice may be made between Inorganic and Organic Chemistry. By this division of the subject a student wishing to apply himself specially to the inorganic side of the science, may attend during his second year the Honours course in Metals, and courses on Geology or Mineralogy, and during his third year, courses on Metallurgy and on Geology or Mineralogy; while a student wishing to apply himself specially to the organic side of the science, may attend during his second and third years the Courses on Organic Chemistry, and courses on the Coal Tar Colours and on Dyeing and Printing.

Part of the Laboratory practice in the second and third years will consist in the examination and analysis of raw materials, products from chemical works, &c., in connection with the special courses of lectures on Applied Chemistry. In the Chemistry and Physical laboratories the practical work in the second year will be arranged in accordance with the branch of Chemistry selected by the candidate.

In the third year the student, if sufficiently advanced, will be set to work on some analytical process or problem in Applied Chemistry, under the direction of the teaching staff.

ELECTRO-CHEMICAL LABORATORY.

In the new Laboratories there is a complete equipment for Electro-chemical and Electro-metallurgical work. **Demonstrator and Assistant Lecturer in Electro-technics,** Robert Beattie, B.Sc. (Durham). **Demonstrator and Assistant Lecturer in Electro-chemistry,** R. S. Hutton, M.Sc. (Vi&.). **Assistant Demonstrators,** James Lord, B.Sc. (Vi&.); R. C. Wale, B.Sc. (Vi&.).

For further particulars apply to the Registrar, Mr. S. Chaffers.

UNIVERSITY COLLEGE, NOTTINGHAM.

DEPARTMENTS OF CHEMISTRY AND METALLURGY.

Professor of Chemistry—F. Stanley Kipping, Ph.D., D.Sc., F.I.C., F.R.S.

Demonstrators of Chemistry—R. M. Caven, D.Sc., F.I.C.; G. D. Lander, D.Sc.; and G. Sand, Ph.D., M.Sc.

The Classes of the College are open to students of both sexes above sixteen years of age.

The Session commences on October 6th, for both Day and Evening Classes.

Lecture Courses.—The Chemistry Day Lectures extend over three years. In the first year a student enters for the course on Elementary Inorganic Chemistry. In his second year he attends Lectures on both Inorganic and Organic Chemistry. In his third year he attends courses

on Advanced Organic Chemistry, Physical Chemistry, and Advanced Inorganic Chemistry.

The fees for the Day Lectures and Classes are as follows: First year, two Lectures per week, 15s. per term. Second year, three Lectures per week, 22s. 6d. per term. Third year, four Lectures per week, 30s. per term.

Demonstrations and Lectures on Analytical Chemistry are given, and Chemical Calculation and Tutorial classes are also held. Various short courses of lectures on Electrochemistry and other special subjects are delivered during the Session.

Students may qualify themselves by attendance at these lectures and classes for the Examinations of the Universities of London, Cambridge, or Oxford, and for the Medical Examinations of the Royal College of Surgeons and of the Universities of Cambridge and Edinburgh: they may also obtain instruction in Chemistry for technical or other purposes, and can enter for a full Chemical Engineering Curriculum. Special attention is given to the requirements of candidates for the Associateship of the Institute of Chemistry.

Practical Chemistry and Metallurgy.—The Chemical and Metallurgical laboratories are open every day from 9 to 5, except on Saturday, when the hours are from 9 to 1; also on Tuesday and Thursday evenings from 7 to 9. Each Student works independently of other Students at a course recommended by the Professor. Instruction is given in general Chemical Manipulation, in Qualitative and Quantitative Analysis, and in the methods of Original Chemical Investigation and Research; Students are also enabled to work out the applications of Chemistry to Pharmacy, Metallurgy, Dyeing, Agriculture, Brewing, Iron and Steel, Tanning, and other Manufacturing Processes. Fees for day students: For one term, £7; for the session, £18; for six hours weekly 40s., and 5s. extra for each additional hour per week. For evening students, 10s. for two hours per week, three hours 15s., four hours 20s., per term.

Research Work.—Students or others wishing to undertake research work in pure or Applied Chemistry will be afforded every facility for doing so and may be admitted at reduced fees. The Laboratories are fully equipped with apparatus and chemicals necessary for such work.

Courses of Technical Chemistry Lectures are also given on Engineering, Dyeing and Bleaching, Brewing, Plumbing, Bread-making, Gas Manufacture, and on other processes of applied Chemistry.

Pharmaceutical Students are provided with Lectures and Laboratory work suitable for the preparation for the Minor and Major Examinations. Special lectures will also be given in Pharmacy, Materia Medica, and all other Pharmaceutical subjects.

Evening Classes.—Evening Lectures and Laboratory instruction will be given in Pure and Applied Chemistry, and the laboratories are open for practical work on Tuesday and Thursday evenings from 7 to 9. Fee for each Lecture Course, 5s.; for each Laboratory Course, 10s.

Full information concerning all College Classes is given in the College Prospectus, price one penny.

UNIVERSITY COLLEGE, SHEFFIELD.

Professor of Chemistry—W. Carleton Williams, B.Sc., F.C.S.

Lecturer—G. Young, Ph.D., F.R.S.E.

The Session will commence on October 1st.

Matriculation Course.—Tuesday and Friday 10 to 11. Fee, £2 2s.

Intermediate Course.—Inorganic Chemistry. Monday and Thursday from 10 to 11 a.m. £2 12s. 6d.

Organic Chemistry.—Elementary: Saturdays, 10 to 11; fee, £1 11s. 6d. Honours: Tuesdays and Thursdays, 12 to 1; fee, £2 12s. 6d. Advanced: Thursdays, 4 to 5; fee, £1 11s. 6d. Special Course: Hours to be arranged; £1 1s.

Physical Chemistry.—Tuesday, 11 to 12. Fee, £1 11s. 6d.

Chemical Philosophy.—Thursday, 11 to 12. Fee, £1 11s. 6d.

A Course of Lectures is arranged for Medical Students, with a special class in Qualitative Analysis.

Laboratory.—Working hours to be arranged between Professor and Students.

Sessional Fees for Day Students:—Six hours per week, £5 5s.; Nine, £7; Twelve, £8 8s.; Eighteen, £11 5s.; Twenty-four, £14; Thirty-two, £17.

Day Students may not enter for less than six hours a week. Students joining the Laboratory at Christmas will be charged two-thirds and at Easter one-third of the Fees for the whole Session.

Fees for short periods (working thirty-two hours per week):—For one month, £3 3s.; two months, £5 5s.

A course of Elementary Practical Physics and Practical Chemistry, which will meet the requirements of candidates for the Diploma of Public Health and for the Licentiate of Sanitary Science, will be held during the Michaelmas and Lent terms. Fee £5 5s.

An arrangement has been entered into with the Science and Art Department, South Kensington, which will enable Science Teachers to work in the Chemical Laboratory for three, six, or twelve hours a week on payment of one-quarter of the usual fee, the Department being willing to pay the remainder under certain conditions, of which full information may be obtained on application to the Registrar.

Evening Classes.—Lectures, Wednesday, 8 to 9. Laboratory instruction, Wednesday, 6 to 9. Sessional Fee, Lecture Class and Laboratory, on Wednesday evening, £1 10s. Fee for one term, 17s. 6d.

DEPARTMENT OF METALLURGY.

Professor of Metallurgy—J. O. Arnold. This Department has been equipped to meet the requirements of the local industries. The Laboratory is fitted with the most modern apparatus for metallurgical analysis, more especially with appliances for the rapid and accurate chemical examination of Iron and Steel, Fuel, and Refractory materials. It also contains a complete pyrometric installation, and a new laboratory for the study of the micrographic analysis of metals has been completed and fully equipped with specially designed microscopes by Ross, polishing tables, etching appliances, incandescent light for evening work, &c. The School is now the most complete of its kind for teaching the practical manufacture, the chemical constitution, and the physical properties of Steel. Special attention is given to the determination of the microscopic constituents of Steel. Although the chief industry of the district occupies the central position in the course of instruction, general metallurgy is not neglected, but is dealt with in a separate syllabus, dealing with metals (other than iron and steel) used in the arts. Students are thus enabled to select and at once enter upon a course of scientific metallurgical training of immediate practical utility. They may take up and work through any portions of the course, but certificates will be granted only to those who follow the prescribed courses, and pass the necessary examinations. Lectures on Iron and Steel Manufacture, on Fuel and Refractory Materials, and on General Metallurgy. Practical Metallurgy:—Laboratory, Furnaces, Foundry, and Testing Machine Course; Practical Course of Metallurgy other than Iron and Steel; Practical Fuel Course for Students engaged in Collieries or Gas-Works.

A new steel works is now being erected in connection with the Sheffield College at a cost of about £10,000. The works are designed to include a two ton open-hearth furnace, a one ton Bessemer plant, and a four-hole crucible melting plant. The works will also be provided with a hammer and rolling mill capable of dealing with four-inch ingots. Differential annealing furnaces, oil-brightening and lead-tempering tanks will constitute part of the new plant. Additional laboratory accommodation will be attached to the works, viz., chemical laboratory for staff, chemical laboratory for post graduate students, together with a complete magnetic laboratory for hysteresis, &c.

UNIVERSITY COLLEGE, DUNDEE.

UNIVERSITY OF ST. ANDREWS.

Professor of Chemistry—James Walker, Ph.D., D.Sc., F.R.S.*Assistant Lecturers*—J. S. Lumsden, Ph.D., D.Sc., and J. K. Wood, D.Sc.*Lecture Assistant and Laboratory Steward*—J. Foggie, F.C.S.

The Winter Session begins on October 14th, and ends on March 18th. The Summer Session extends from the middle of April to the end of June.

The *Junior Lecture Course on Systematic Chemistry* is given daily during the Winter Session, and embraces the Elements of Inorganic and of Organic Chemistry.

Advanced Courses, of about fifty lectures each, will be given during the year as follows:—

Organic Chemistry; Inorganic Chemistry, including the more important technological applications; Theoretical and Physical Chemistry; Bleaching and Dyeing, including the Chemistry of the Textile Fibres.

Practical Instruction in all of the above branches will be given in the Laboratories and Dye-house. Special facilities are afforded to Research Students.

UNIVERSITY OF ABERDEEN.

CHEMISTRY.

Professor—Francis R. Japp, M.A., LL.D., F.R.S.*Demonstrators*—W. B. Davidson, M.A., D.Sc., and F. W. Gray, M.A., B.Sc.

I. *General Lecture Course (100 Lectures)*.—Daily during the Winter Session. The subjects treated of include (1) The Laws of Chemical Combination and the General Principles of Chemistry; (2) Non-metallic and Metallic Elements, and their Compounds; (3) Organic Chemistry; (4) Applications of Chemistry to the Arts and Manufactures. Fees, for first attendance, £3 3s.; for subsequent attendance, £2 2s. A Tutorial Class (without fee), conducted by the Senior Demonstrator, is held in connection with this course.

II. *Special Lecture Course on Organic Chemistry (50 Lectures)*.—Daily during the Summer Session. Fee, £2 2s.

III. *Practical Course for Medical Students*.—This course, which occupies five hours a week during the Summer Session—in all fifty hours—is devoted to practice in Elementary Qualitative Analysis. Fee, £3 3s.

IV. *Chemical Laboratory*.—The Laboratory is open to Students daily from 9 a.m. to 5 p.m. Each Student on entering is allowed to arrange his hours of work so as to suit his own convenience, but must adhere to these hours when once fixed. The Laboratory instruction includes: (1) General experiments; (2) Preparations; (3) Qualitative analysis; (4) Quantitative analysis: gravimetric, volumetric, and gasometric. Fee, £3 3s. a session. A certain number of free places are available, on the recommendation of the Professor, and subject to the approval of the Senatus, for research students.

In connection with the Laboratory work, three Lectures a week on Physical Chemistry and selected portions of Inorganic Chemistry are delivered during the Winter Session by the Lecturer, Dr. W. B. Davidson. Fee, £2 2s.

*Agricultural Chemistry.**Lecturer*—James Hendrick, B.Sc., F.I.C.

This course is given during the Winter Session, and includes both Lectures and Laboratory work. The Lectures deal with the chemistry of the atmosphere, the soil, manures, and foods. The physiological chemistry of plants and animals, the composition and manurial requirements of crops, and dairy chemistry, are also treated of. The Laboratory work is primarily intended to accompany and illustrate the Lectures. Exercises dealing with the properties and composition of soils, manures, feeding stuffs and waters, and with the impurities and adulterations of these, occupy much of the time given to practical work. The fee for the whole course—lectures and practical work—is £3 3s.

UNIVERSITY OF EDINBURGH.

DEPARTMENT OF CHEMISTRY.

Professor—Alex. Crum Brown, M.D., D.Sc., F.R.S.*Lecturers*—L. Dobbin, Ph.D.; H. Marshall, D.Sc.; and W. W. Taylor, M.A., D.Sc.

The working terms are—Winter Session, from middle of October to middle of March; Summer Session, from beginning of May to middle of July.

Lecture Courses.—During the Winter Session a General Course of Chemistry for medical and science students is given by the Professor. The class meets daily; fee, £4 4s. An Advanced Course of twenty-five lectures is also given in the Winter Session; fee, £2 2s. An Intermediate Course is held in summer; fee, £2 2s. There is also a class on Chemical Theory, by Dr. Dobbin; fee, £1 1s.; and a class on Mineralogy and Crystallography, by Dr. Marshall; fee, £2 2s. All these Lectures, except the General Course, are now open to women.

In addition to the above, Lecture Courses are from time to time given by the Assistants on particular branches of Organic and Inorganic Chemistry. These Lectures are free to Laboratory Students.

Tutorial classes are held in connection with the General Course.

Laboratories.—Practical classes for Medical Students meet daily during the latter part of the Winter Session and in the Summer Session. (Fee, £3 3s.) The laboratories for analytical and advanced practical work are open daily from 9.30 till 4.30. (Fees: Whole Day—Winter Session, £10 10s.; Oct.-Dec., Jan.-March, or Summer Session, £5 5s. Half Day—Winter Session, £6 6s.; Oct.-Dec., Jan.-March, or Summer Session, £3 3s. Preference will be given to students in the above order. Students who are not Matriculated may attend the Chemical Laboratory on payment of the entrance fee of 5s. in addition to the Laboratory fees. Full Courses of instruction are given in Analytical, Practical Organic and Inorganic Chemistry, including Gas Analysis, Metallurgy, and Assaying. Facilities are afforded to advanced students who desire to undertake chemical investigations.

Various prizes and scholarships are attached to the laboratory and general class.

Graduation.—Two Degrees in Pure Science are conferred, viz., Bachelor of Science (B.Sc.) and Doctor of Science (D.Sc.).

Candidates for Degrees in Science, if not graduates (by examination) in Arts in one of the Universities of the United Kingdom or in a Colonial or Foreign University recognised for the purpose by the University Court, must pass a preliminary examination in (1) English; (2) Latin, Greek, French, or German; (3) Mathematics; (4) One of the languages Latin, Greek, French, German, Italian, not already taken under (2), or Dynamics. In the case of a student whose native language is other than European, the Senatus may, at the Preliminary Examination, accept such language as a substitute for a modern European language. The Senatus may also in such a case accept as an alternative to Latin or Greek any other classical languages, such as Sanscrit or Arabic.

The First B.Sc. Examination embraces Mathematics, or Biology (*i.e.*, Zoology and Botany), Natural Philosophy, and Chemistry. The Final B.Sc. Examination includes any three or more of the following subjects:—Mathematics, Natural Philosophy, Astronomy, Chemistry, Human Anatomy, including Anthropology, Physiology, Geology, including Mineralogy, Zoology, including Comparative Anatomy, and Botany, including Vegetable Physiology. In the Final Examination two written papers are set in each subject professed, the second of a higher standard than the first. Candidates must pass the first section in all, and the second section in at least one, of the subjects professed; the same regulations apply also to the Practical and Oral Examinations. Chemistry in this examination embraces Inorganic, including Mineralogical, Chemistry; Organic Chemistry; Chemical Crystallography; History of Chemistry. In the written papers a

choice of questions is allowed, so as to adapt the examination to the various courses of advanced study which candidates may have selected. Practical Examination:—Complex Qualitative Analysis; Inorganic Preparations; Gravimetric and Volumetric Analysis. Each candidate taking the higher standard will also be examined on Organic Preparations, Ultimate Organic Analysis, and one of the following subjects, selected by himself:—Gas Analysis; Assaying; Physico-chemical Measurements.

A candidate for the D.Sc. Degree must submit a thesis on original work done by him. The Thesis must be approved before the candidate is allowed to proceed to Examination. The candidate in Chemistry may be required to pass a searching examination in one of the following branches:—(1) The Chemistry and Chemical Technology of Inorganic Bodies, including Metallurgy; (2) Organic Chemistry; and to show a thorough practical acquaintance with chemical analysis in all its branches, and with the preparation of pure substances.

HERIOT-WATT COLLEGE, EDINBURGH.

Principal—A. P. Laurie, M.A., D.Sc., F.R.S.E.

Professor—John Gibson, Ph.D., F.R.S.E.

Assisted by the following Lecturers and Demonstrators:

—Archibald Boon, B.A., B.Sc.; Andrew F. King, F.I.C.; and J. P. Longstaff, B.Sc.

The Session begins September 29th, 1902.

The curriculum of this College comprises both Day and Evening Classes, each department providing the higher general and technical education.

The Chemistry Course is designed to meet the wants of Manufacturing Chemists. The instruction consists of Courses in Chemistry, Physics, Mathematics, Engineering, and Mechanical Drawing, with Special Courses in Gas and Paper Manufacture, Brewing, &c., by Experts, in the third year. Special attention will be paid to Electro-chemistry. The Chemistry Classes are recognised by the University as qualifying for the B.Sc., in Chemistry, and are also recognised by the Institute of Chemistry.

Matriculation Fee, 5s.; Composition Fee for Engineering Courses, £10 10s.; for Chemistry Courses, £10 10s., £12 12s., £15 15s. Full particulars are published in the Calendar of the College early in September.

GLASGOW AND WEST OF SCOTLAND TECHNICAL COLLEGE.

Professor of Chemistry—G. G. Henderson, D.Sc., M.A.

Professor of Metallurgy—A. Humboldt Sexton, F.C.S., F.R.S.E.

Also, Professors and Lecturers in the other leading branches of Pure and Applied Science and Technology.

The main objects of this College are to afford a suitable education to those who wish to qualify themselves for following an industrial profession or trade, and to train teachers for technical schools. It was founded by an Order in Council, dated 26th November, 1886, according to a scheme framed by the Commissioners appointed under the provisions of the Educational Endowments (Scotland) Act, whereby Anderson's College, the Young Chair of Technical Chemistry in connection with Anderson's College, the College of Science and Arts, Allan's Glen's Institution, and the Atkinson Institution were placed under the management of one governing body.

The Diploma of the College is awarded to Day Students who have attended prescribed courses of instruction and passed the necessary examinations. The ordinary courses extend over three years, but arrangements are made for advanced students continuing their studies in special departments.

Complete courses of instruction are provided in both Day and Evening Classes.

Copies of the Calendar for 1902-1903 may be had from Mr. H. F. Stockdale, the Secretary, 38, Bath Street, Glasgow, price by post, 1s. 4d. Prospectuses will be sent free.

UNIVERSITY OF ST. ANDREWS.

UNITED COLLEGE OF ST. LEONARD AND ST. SALVATOR.

Professor of Chemistry—T. Purdie, B.Sc., Ph.D., LL.D., F.R.S.

The Session begins on October 14th. A Competitive Examination, open to intending Students of Arts, Science, and Medicine, for about forty-three Entrance Bursaries, ranging in value from £40 to £10 each per annum, will be held on September 26th and following days. Twenty-six of these Bursaries are restricted to Men, three to Men or Women, and about fourteen to Women, the latter being intended for women who at the conclusion of their Arts or Science Course will proceed to Medicine.

A Hall of Residence is provided for Women Students.

Two Degrees in Science are conferred by the University of St. Andrews, viz., Bachelor of Science (B.Sc.) and Doctor of Science (D.Sc.), and Chemistry is also included in the curriculum for the M.A. Degree; the regulations will be found in the "University Calendar."

Lecture Courses.

Two distinct Courses of Lectures are given, each comprising at least one hundred meetings of the class.

First Year's Course.—This Class meets at 11 o'clock on five days in the week. The introductory lectures treat of the Nature of Chemical Action, the Classification of Substances into Elements and Compounds, the Phenomena of Oxidation, and the Composition of Air and Water. The Laws of Chemical Combination and the Atomic Theory are next discussed, after which the more commonly occurring elements and inorganic compounds are described systematically. Elementary Organic Chemistry is also included in the Course.

The chemistry of manufactures is referred to only cursorily; special attention, on the other hand, is given to those parts of the science which are of general educational value, and as much of the theory of chemistry is introduced as is compatible with elementary treatment.

This course of instruction is intended to meet the requirements of the M.A., First Science, and M.B., Ch.B. Examinations, so far as Theoretical Chemistry is concerned.

Second Year's Course.—The first part of the Course is devoted to Organic Chemistry, and the second part to General and Physical Chemistry, the instruction in general being such as is required for the Second B.Sc. Exam.

Certificates are awarded on the results of examinations, and the "Forrester Prize" of about £10 is awarded to the best Student of the year.

Fee for the Session, for each Course, £3 3s.

Practical Chemistry.

The Laboratory is open daily from 9 a.m. to 4 p.m., except on Saturdays, when it is closed at 1 p.m. The work pursued in the Laboratory comprises:—(1) The performance of experiments illustrative of the Principles of Inorganic and Organic Chemistry; (2) Qualitative and Quantitative Analysis; (3) Original Investigation. Each student pursues an independent course of study under the supervision of the Professor or Demonstrator, the nature of the work varying with the proficiency of the student and the particular object he may have in view. Suitable courses of instruction in Practical Chemistry are provided for candidates for the Examinations in Arts, Science, and Medicine.

The fee for each course of Practical Chemistry is £3 3s. A certain number of working places in the Laboratory will be available without fee for students who are capable of undertaking original investigation.

QUEEN'S COLLEGE, BELFAST.

Professor—E. A. Letts, Ph.D., D.Sc., F.R.U.I., F.R.S.E., &c.

I.—Chemistry.—The lectures are delivered at 3 p.m., on the first five days of each week, and terminate at the end of March. The course is divided into three parts:—

(1) Chemical Philosophy; (2) Inorganic Chemistry; (3) Organic Chemistry. Fee, £2.

II.—*Practical Chemistry*.—In this course the Students are instructed in the general methods of conducting Chemical Analyses. Fee, £3.

III.—*Laboratory Pupils*.—The Chemical Laboratory is open from November until the end of March, and from May 1st until the third week of July, on the first five days of the week, from 10 a.m. until 4 p.m. Students are admitted as working pupils on payment of a fee of £5 for the first period, or of £3 10s. for the second period (or for a single term).

The new Chemical Laboratories are now open, and are provided with all modern appliances. Special facilities are offered to those engaged in Research work.

Scholarships.—The following College Scholarships are awarded specially in connection with the schools of Chemistry and Physics:—Senior Scholarships in Chemistry alone awarded annually of £40, and tenable for one year. Andrews Studentship in Chemistry and Physics, value about £80 annually, and tenable for two years. 1851 Exhibition Scholarships: One of these Scholarships has hitherto been placed at the disposal of the College every two years, of the annual value of £150, tenable for two or under special conditions for three years.

The following Scholarships, &c., of the Royal University of Ireland are awarded in the same group of subjects (Chemistry and Physics):—At the B.A. Degree examination, First-class Exhibitions of £42 each and Second-class Exhibitions of £21. At the M.A. Degree examination, a Studentship of £100 per annum, tenable for three consecutive years. Also a Junior Fellowship, tenable for four consecutive years, of the annual value of £200.

QUEEN'S COLLEGE, CORK.

Professor—Augustus Edward Dixon, M.D.

Demonstrator—Robert Elliott Doran, F.C.S.

The College Session will commence on October 21st, 1902, and end on June 13th, 1903. All classes are open to male and female students.

The courses in Chemistry, though designed primarily to meet the requirements of candidates proceeding to the Examinations in Arts, Medicine, and Engineering of the Royal University of Ireland, of the Medical Licensing Bodies of Dublin and Edinburgh, and of the Pharmaceutical Society of Ireland, can be specially arranged as required, so as to render them suitable to any particular course of study. The following courses are provided:—

Systematic Chemistry.

I. General Lecture Course; three terms. Inorganic Chemistry, Elementary Organic Chemistry, and Chemical Philosophy.

II. Advanced Organic Chemistry, and Chemical Philosophy.

Practical Chemistry.

III. Winter Course of Practical Analytical Chemistry; commencing early in January, 1903.

IV. Summer Course of three months' duration, for students of Medicine; ending about the third week in June.

V. Course for Pharmaceutical Students; held during the second and third terms.

Laboratory Work.

VI. The Chemical Laboratory is open daily from 10 to 4 o'clock, to Students entering for Special Courses of practical work in General Inorganic Chemistry, Qualitative and Quantitative Analysis, Organic Chemistry, or for the purpose of Original Investigation.

Scholarships, &c.—In addition to the Class Prizes given at the Sessional Examinations, the College awards a Senior Scholarship of the value of £40, for Chemistry and Physics (either of which may be taken as a limited course), and numerous Junior Scholarships in Arts, Medicine, and Engineering, of which chemistry forms a part, value from £20 to £24 each, annually. The Royal University of Ireland also offers Exhibitions in Chemistry and Physics,

First-class value £42, and Second-class value £21, together with other prizes, e.g., a Studentship of £100 per annum, tenable for three years; and the 1851 Exhibition Scholarships, value £150, tenable for two, and, under certain circumstances, for three years, have from time to time also been placed at the disposal of the College.

Full particulars as to Lectures, Fees, Scholarships, Exhibitions, &c., are contained in the Regulations extracted from the College Calendar, which will be supplied on application to the Registrar.

QUEEN'S COLLEGE, GALWAY.

Professor—Alfred Senior, Ph.D., M.D., F.I.C., F.C.S.

Demonstrator—William Goodwin, F.C.S.

The College Session commences in October and ends in June.

Chemistry is studied by attendance at Lectures, by work in the Laboratories, and by the use of the College Library. The Courses in the several faculties are arranged with a view to the requirements of the Royal University of Ireland, but are adapted also to those of other Universities and licensing bodies.

Lecture Courses. Faculty of Arts.—1. Second year's Course, Inorganic and the Elements of General Chemistry. 2. Third year's Course, Advanced Organic Chemistry. 3. Fourth year's Post-Graduate Course, Advanced General Inorganic and Organic Chemistry. *Faculty of Medicine.*—First year's Course, Inorganic and Elementary Organic Chemistry. *School of Engineering.*—First year's Course, Inorganic Chemistry.

Laboratory Courses. Faculty of Arts.—1. Second year's Course, Exercises in Inorganic Qualitative Analysis. 2. Third year's Course, Quantitative Analysis and other experiments to suit the requirements of individual Students. 3. Fourth year's Post-Graduate Course, Advanced Quantitative Analysis, Organic and Inorganic Preparations, and determination of their Physical and Chemical characters. 4. The Laboratories are also open to Students for work in other branches of Chemistry. *Faculty of Medicine.*—1. Second year's Course, Inorganic and Organic Elementary Qualitative Analysis, and the Chemical Examination of Urine. *School of Engineering.*—1. Second year's Course, Inorganic Qualitative Analysis.

A Scholarship in Chemistry is offered for competition each year of the value of £40, and Chemistry enters into the subjects required for numerous Scholarships in Arts, Medicine, and Engineering. The Royal Commissioners for the Exhibition of 1881 offer a Scholarship of £150 per annum. This Scholarship has just been awarded for the third time to a Chemistry Student of this College, and to another Student it has been renewed for a third year.

For details as to Fees, Regulations as to Scholarships, and other particulars apply to the Registrar, from whom the Calendar, published in December, and the Extracts from Calendar, published in advance in July, may be obtained.

ROYAL COLLEGE OF SCIENCE, DUBLIN.

Professor of Chemistry—W. N. Hartley, F.R.S.

Assistant Chemist—J. Holms Pollok, B.Sc.

Demonstrator of Chemistry and Assaying—A. E. B. Manders.

The Royal College of Science supplies, as far as practicable, a complete course of instruction in Science applicable to the Industrial Arts, and is intended also to aid in the instruction of teachers for the local Schools of Science.

Diplomas are awarded in the Faculties of Engineering, and Manufactures, Physics, and Natural Science. If accompanied by a certificate from the Professor of Chemistry, the Diploma of Associate of the Royal College of Science in the Faculty of Manufactures is recognised by the Council of the Institute of Chemistry of Great Britain and Ireland as qualifying candidates for admission to the practical examinations of the Institute.

The instruction in Chemical Science includes (1) General Chemistry; (2) Advanced Chemistry, including Organic

Chemistry; (3) Analytical and Experimental Chemistry; (4) Instruction in Chemical Research.

Fees payable by Non-Associate Students:—£2 for each separate Course of Lectures. For Analytical Chemistry and Research—£5 for three months; £9 for six months; £12 for the entire session. For Assaying—£5 for three months; £9 for six months; £12 for the entire session.

The following are supplementary courses of instruction:—

- (1) Laboratory Instruction in the Theory of Chemistry.
- (2) An Analytical Course for Students in Engineering.
- (3) A Course of Practical Chemistry for Medical Students.
- (4) The Analysis of Water, Air, Food, and Drugs, intended for the instruction of Public Analysts and Medical Officers of Health.
- (5) A Course of Chemistry for Pharmaceutical Students.

There are four Royal Scholarships of the value of £50 each yearly, with Free Education, including Laboratory Instruction, tenable for two years; two become vacant each year; they are awarded on the results of their examinations to Associate Students, not being Royal Exhibitioners, who have been a year in the College.

CHEMICAL LECTURES, CLASSES, AND LABORATORY INSTRUCTION.

CITY AND GUILDS OF LONDON INSTITUTE FOR THE ADVANCEMENT OF TECHNICAL EDUCATION.—The operations of the City and Guilds of London Institute are divided broadly into four branches: the educational work of three London Colleges, and of the Technological Examinations. Programmes of the London Colleges may be had on application to the Head Office of the Institute, Gresham College, Basinghall Street, London, E.C., or from the respective Colleges. The Technological Examinations (Examinations Department, Exhibition Road, S.W.), are conducted once every year at various centres throughout the kingdom. Programme, with Syllabus of Subjects, &c., may be obtained of Messrs. Whittaker and Co., Paternoster Square, London, or through any bookseller, price 10d., net. — *City and Guilds Technical College, Exhibition Road.*—Professor of Chemistry, H. E. Armstrong, Ph.D., F.R.S. The object of this Institution is to give to London a College for the higher technical education, in which advanced instruction shall be provided in those kinds of knowledge which bear upon the different branches of productive industry, whether Manufactures or Arts. The main purpose of the instruction given is to practically demonstrate the application of different branches of science to various manufacturing industries. In order that this instruction may be efficiently carried out, the Institution, in addition to the lecture theatres and class rooms, is fitted with laboratories, drawing offices, and workshops; and opportunities are afforded for the prosecution of original research, with the object of the more thorough training of the students, and for the elucidation of the theory of industrial processes. The courses of instruction are arranged to suit the requirements of—1. Persons who are training to become Technical Teachers; 2. Persons who are preparing to enter Engineers' or Architects' offices, or Manufacturing works; 3. Persons who desire to acquaint themselves with the scientific principles underlying the particular branch of industry in which they are engaged. The Matriculation Examinations begin on Tuesday, September 23rd, and the Winter Session opens on Tuesday, October 7th. *City and Guilds Technical College, Finsbury.*—Professor of Chemistry, Raphael Meldola, F.R.S. The operations of the Technical College, Finsbury, are divided into two distinct portions: Day Classes for those who are able to devote one, two, or three years to systematic technical education; Evening Classes for those who are engaged in industrial or commercial occupations in the daytime and who desire to receive supplementary instruction in the application of Science

and of Art to the trades and manufactures in which they are concerned or employed. Each Professor is assisted by Demonstrators. Besides these there are Lecturers and Teachers in special subjects. An examination for the admission of Students will be held at the College on Tuesday, September 23rd. Session begins October 7th. *South London Technical Art School.*—Classes in Modelling, Design, Drawing and Painting, House Decoration.

CITY OF LONDON COLLEGE, White Street, Moorfields.—Courses of Evening Lectures and Laboratory Practice in Chemistry and Physics, conducted by Mr. I. S. Scarf, F.I.C., F.C.S., assisted by Messrs. H. W. Harrie, F.C.S., and C. A. West, B.Sc., &c. Session commences Sept. 29.

BATTERSEA POLYTECHNIC.—Principal, Mr. Sidney H. Wells, Wh. Sc. Inorganic, Organic, and Technological Chemistry, Mr. John Wilson, M.Sc. (Vit.), assisted by Mr. John L. White, M.Sc., Mr. H. G. Wayling, B.Sc., and Mr. B. C. Polkinghorne, B.Sc., F.C.S. Day and Evening Classes in Science and Art subjects. Session opens Sept. 16. The principal work of the Institute is the provision of Evening Classes for both sexes in all subjects of Technology, Pure and Applied Science, Art, Commerce, Domestic Economy, and Music; but it also provides a Technical Day School, a Science Day School for Boys and Girls, a Training School of Domestic Economy, a Domestic Economy School for Girls, a Day School of Art, and Special Day Courses in Science and Technical subjects. During the last Session, 250 Evening Classes in more than 100 different subjects were attended by over 3100 individual students, while 520 students were in regular attendance at the Day Schools and Classes.

BIRKBECK LITERARY AND SCIENTIFIC INSTITUTION, Breams Buildings, Chancery Lane.—Chemistry Courses will be conducted, commencing Monday, September 29, adapted for the Elementary, Advanced, and Honours Examinations of the Board of Education and for various Examinations for the B.Sc. and M.B. Degrees of the London University, by Mr. J. E. Mackenzie, Ph.D., D.Sc. This Institution, which has now completed seventy-nine years of educational work in the metropolis, commences its new Session on Monday, September 29th. The Day and Evening courses of study, comprise the various branches of Natural Science, Mathematics, classical and modern Languages, Economics, Commercial Subjects, Law, Mental Science, Music, and Art. The courses provide for the Examinations of the University of London, in the faculties of Arts, Science, and Commerce, those of the Conjoint Board, Civil Service, &c. The Report for the last session shows that during the year 58 students passed some university examination, while large numbers gained successes at various public examinations. The Institution has had many additions to its appliances in recent years, and the Physical, Chemical, Biological, and Metallurgical laboratories are now very thoroughly equipped. The Day classes provide courses in Chemistry, Biology, Physics, Mathematics, and Geology for the Science Degrees of London University. There are also Day classes in modern Languages, including Russian and Spanish; courses of Lectures on French and German Literature are given both in the afternoon and evening. The School of Art is open both day and evening. A new reading room, and a new magazine room have recently been provided. The Metallurgical laboratory has been extended and reorganised, and classes are held both in the day and evening. The Calendar supplies complete syllabuses of the classes and full information about the Institution. A Lecture or Entertainment is given in the Theatre every Wednesday evening.

BRIXTON SCHOOL OF CHEMISTRY AND PHARMACY, 171, Brixton Road, London.—Principal, Dr. A. B. Griffiths, F.R.S. (Ed.). Lectures on Inorganic and Organic Chemistry, Physics, Botany, &c., for Pharmaceutical, Medical, and other students. The benches in the laboratory are fitted with every convenience. There are courses in Research Work. Full particulars as to fees, &c., may be obtained by writing to the Principal.

BOROUGH POLYTECHNIC INSTITUTE, 103, Borough Road (near the Obelisk).—Chemistry: Evening Lectures and Laboratory Work in Inorganic and Organic Chemistry (Session fees, per course, 8s. to 20s.). Day Practical class in Electro-chemistry, lecture on Friday evenings (70s.) Special Saturday Morning Class in Practical Chemistry (10s.); Dr. F. Mollwo Perkin, assisted by Dr. E. C. Jee and A. Fontana, B.A. Electricity and Magnetism: Evening Lectures and Laboratory Work (Session fees, per course, 8s. to 10s.), Dr. J. Henderson, assisted by H. Saunders. Special Evening Courses on Painter's Oils, Colours, and Varnishes. Session opens Monday, September 22, 1902.

SOUTH WESTERN POLYTECHNIC, Chelsea, S.W.—Principal, Herbert Tomlinson, B.A., F.R.S. Professor of Chemistry, J. B. Coleman, A.R.C.S., F.I.C. Day and Evening Courses in Theoretical and Practical Chemistry and several branches of Applied Chemistry, including Metallurgy, Assaying, &c. Special Classes are held for the Matriculation, Intermediate Science, and B.Sc. Examinations of the University of London. Prospectuses of Day and Evening Classes may be obtained from the Secretary, 1d. each, by post 2½d.

THE GOLDSMITHS' INSTITUTE, New Cross, S.E.—Head of the Chemistry Department, Arthur Lapworth, D.Sc.(Lond.), F.I.C.; Assistants, Mr. A. C. O. Hann and others. Lectures and Practical Classes in General Chemistry and Photography, also in Chemistry applied to other industries, are held in the evenings from 7.15 to 10, and are open to both sexes. Special attention is paid to Technical Laboratory work and the investigation of manufacturing difficulties. Session commences September 22.

EAST LONDON TECHNICAL COLLEGE, Mile End Road, E.—Chemistry: Professor, J. T. Hewitt, M.A., D.Sc., Ph.D.; Assistant Lecturer and Demonstrator, F. G. Pope. Lectures and Practical Classes are conducted in the Day Technical College, the regular College Course being three years. The work of the first year corresponds with the requirements of the Int. Sci., Lond., that of the next two years with the degree examination (B.Sc.). Evening Classes are also held. The Day College opens on Sept. 8. Evening Classes begin Sept. 23.

NORTHERN POLYTECHNIC INSTITUTE, Holloway Road, N.—Head of Chemical Department, Mr. H. Charles L. Bloxam. Assistants, Mr. W. H. Watson, A.R.C.S., Mr. F. E. Grant, B.Sc. Lectures and Practical Classes in Elementary, Advanced, and Honours Inorganic and Organic Chemistry are held in the evenings from 6.30 or 7 p.m. to 9.30 p.m., affording instruction in the Courses for the Examinations of the University of London and the Board of Education. The Classes form a Course of Study extending over five Sessions, and are open to both sexes. The Laboratories are open to those wishing to pursue investigations. A reference library is attached to the Chemical Department. Session begins September 22.

NORTHAMPTON INSTITUTE (City Polytechnic), St. John Street Road, E.C.—Principal, R. Mullineux Walsley, D.Sc., &c. Lecturers in Technical Chemistry: Mr. S. Field, A.R.C.S., and Mr. G. Naylor. Day and Evening Classes in Electro-chemistry, Electro-plating, Electrometallurgy, Electrotyping and Stereotyping. (For full details see Prospectus).

CARPENTERS' COMPANY TECHNICAL INSTITUTE, Jupp Road, Stratford, E.—Principal, William Ping, F.C.S. Evening Classes in Theoretical and Practical Chemistry, and in many other Departments of Science and Art. Day Technical School.

UNIVERSITY TUTORIAL COLLEGE, 32, Red Lion Square, Holborn, W.C.—This Institution contains large Laboratories for Chemistry, Physics, Geology, and other subjects. Classes are specialised for Examinations of London University, but students may take up work for any Examination. The special feature of the College is that work goes on all the year round, thus affording students residing in the Country an opportunity of doing practical work during their Vacations.

IMPERIAL COLLEGE OF CHEMISTRY AND PHARMACY, 49 and 51, Imperial Buildings, Ludgate Circus.—Mr. F. Davis, B.Sc. An especial course of instruction is given at this College in Pharmacology and Therapeutics, *vs* the Institute of Chemistry Examination. Students attending the College visit technical works.

WEST HAM MUNICIPAL INSTITUTE.—Principal, Albert E. Briscoe, B.Sc. (Lond.), &c. Chemical Department, Harold A. Auden, M.Sc., &c., assisted by F. H. Streatfeild and H. V. Capsey. Day and Evening Classes.

SOUTH-EASTERN AGRICULTURAL COLLEGE, Wye, Kent.—Chemistry: Lecturer, E. J. Russell, D.Sc. (Lond.); Demonstrators, F. J. Holbrook and F. J. Flymen. Session begins October 1. The courses of instruction in Chemistry are suitable to students intending to become analysts or assistants in works manufacturing manures, feeding-stuffs, &c. The College possesses new and well-equipped Laboratories for Chemistry, Botany, and Bacteriology. The farm, gardens, and experiment grounds afford material for investigation and research. Fee: £8 6s. 8d. per term admits to all Lecture Courses, &c.

BLACKBURN MUNICIPAL TECHNICAL SCHOOL.—Chemistry: Mr. Robert H. Pickard, D.Sc.(Lond.), &c., assisted by Mr. W. H. Duckworth and Mr. Jos. Yates. Session opens Monday, September 15. Full details of the Classes are given in the "Students' Handbook," which may be had at the Institution (price 1d., by post 2½d.).

REDRUTH SCHOOL OF MINES, CORNWALL.—Principal, J. Paul de Castro, M.A. (Cantab.), A.I.C., F.C.S., assisted by a staff of Mining Engineers and Mine Surveyors. Courses of Practical and Theoretical instruction are given in Inorganic Chemistry, Assaying, Mineralogy, and Blow-pipe Analysis, for intending Assayers and Mineral Chemists. Shorter Courses are also held in the above subjects to meet the requirements of intending Mining Engineers, and Prospectors. Practical instruction in Mining given at Pednandrea, the School Mine. Syllabus on application to the Registrar. Session commences Wednesday, September 10.

INSTITUTE OF PUBLIC HEALTH AND TECHNOLOGY, Seymour House, Old Trafford, Manchester.—Principals, Charles Estcourt, F.I.C., F.C.S., &c., and Philip Anderson Estcourt, Ph.D. The course of instruction is especially intended for those who require a knowledge of Chemistry and Bacteriology bearing upon Sanitary matters. Chemical Technology is specially taught with a view to enable Medical Men and others engaged in Sanitary matters to understand the operations at the various works which may come under their control. Persons who desire to take up the branch of Chemical Engineering, including Electrical Engineering, will also have special opportunities of pursuing such studies.

THE MUNICIPAL SCHOOL OF TECHNOLOGY, Sackville Street, Manchester.—This fine building, which has been in partial occupation during the past session, is to be formally opened by the Prime Minister on October 15 next. It occupies an area of upwards of 6800 square yards, and is six floors in height. Its equipment, which is nearly complete, is on a scale of great magnitude. It comprises laboratories and workshops for Mechanical, Electrical, and Hydraulic Engineering, for the Textile industries, and for the industries included within the scope of Architecture. The provision for the teaching of Chemistry, as might be expected in a district where the Chemical industries are important, is of an exceptionally complete character. It comprises, in addition to ample accommodation for pure Chemistry, laboratories for Metallurgy, Brewing, Bleaching, Dyeing and Printing, Paper-making, and Electro-chemistry. The Bleaching, Dyeing, Printing and Finishing, and the Paper-making industries are in addition equipped in a separate building with machinery and appliances on an industrial scale. A Brew-house is also equipped on the low gravity principle, with a plant of four bushel capacity.

LEEDS TECHNICAL SCHOOL (in connection with the Leeds Institute of Science, Art, and Literature), Cookridge Street, Leeds.—Head Master, R. E. Barnett, B.Sc. (Lond.), A.R.C.S. Evening Classes are held in 40 subjects of Science and Technology, among which are:—Chemistry, Inorganic and Organic, Theoretical and Practical, by Mr. R. E. Barnett, B.Sc., A.R.C.S., Mr. J. B. Murray, and Mr. T. R. White, B.A.; Metallurgy, Theoretical and Practical, and Iron and Steel Manufacture, by Mr. B. A. Burrell, F.I.C., F.C.S.; Magnetism and Electricity, and Practical Physics, by Mr. J. E. Tindall, B.A., B.Sc.; Botany, Lectures and Practical Work, by Mr. N. Walker; Photography, by Mr. S. E. Bottomley. Fees:—Lectures, from 2s. 6d. per Session; Laboratory work, from 2s. 6d. Systematic courses arranged for Pharmaceutical Students and for those engaged in Chemical Industries at composition fees. Session commences Sept. 15th. Prospectuses of various Classes free on application to the Secretary, Mr. Arthur Tait. Calendar of the Institute (164 pp.) post free 5d.

WOLVERHAMPTON MUNICIPAL SCIENCE AND TECHNICAL SCHOOL.—Inorganic and Organic Chemistry, Mr. W. Whitehouse, F.C.S.; Physics, Messrs. W. Whitehouse, W. Rogers, and H. J. Tench; Botany, Mr. Sidney Phillips, Ph.C.; Latin, Mr. F. G. Griffiths, M.P.S. Day Classes in Chemistry and Physics, and Evening Classes in Chemistry, Physics, Botany, French, Latin, &c. Special arrangements are made for the requirements of Pharmaceutical and Medical Students. The School is recognised by the Examining Board of the Royal College of Surgeons and Physicians, as a centre for preparation in Chemistry and Physics. Session commences Monday, Sept. 15th. For other particulars and programme, apply Mr. G. F. Chell, Secretary.

CHEMISTRY AT THE BRITISH ASSOCIATION.

THE President of Section B, Professor E. DIVERS, M.D., F.R.S., will deal with some aspects of the Atomic Theory in his address to the Section. The following papers, amongst others, will be read:—

- "Our present Knowledge of Aromatic Diazo-compounds," by Dr. G. T. Morgan.
 - "Reduced Benzene Derivatives containing a Single Nucleus," by Dr. A. W. Crossley.
 - "Present Synthetical Research on the Glucosides," and "The Synthetical Action of Enzymes," by Dr. E. F. Armstrong.
 - "The Alkylation of the Sugars," by Professor T. Purdie, F.R.S., and Dr. Irvine.
 - "The Colour of Iodine-containing Compounds," by Miss Ida Smedley.
 - "On Zirconium Hydrate and other Colloids from Elements of the Fourth Group," by Dr. J. H. Gladstone, F.R.S., and Mr. W. Hibbert.
 - "On some Optical Properties of Tellurium," by Dr. J. H. Gladstone, F.R.S.
 - "The Telluric Distribution of the Elements in relation to their Atomic Weights," by Mr. W. Ackroyd, F.I.C.
 - "The Undesirability of establishing Standard Analytical Methods," by Mr. B. Blount, F.I.C.
 - "The Action of Distilled Water on Lead," by Dr. F. Clowes.
 - "The Decomposition of Urea," by Dr. C. E. Fawcitt.
 - "On the Corrosion of Copper by Sea-water and on the Detection of Traces of Impurity in the Commercial Metal," by Dr. E. A. Letts.
 - "On Experiments to Ascertain the amount of Carbonic Anhydride Absorbed from Sea-water by Air," by Dr. E. A. Letts and Mr. W. Caldwell.
 - "On the Absorption of Ammonia from Water by Algae," by Dr. E. A. Letts and Mr. J. S. Jotson.
- Professor D. Vorländer, of Halle, and Professor E. Knoevenagel, of Heidelberg, are expected to be present as guests of the Association.

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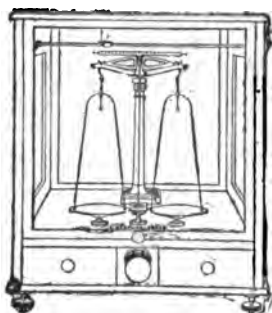
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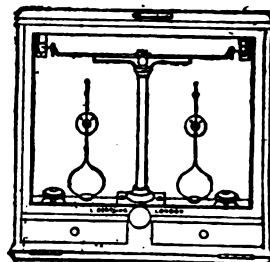


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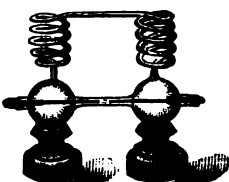
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INAUGURAL ADDRESS OF THE PRESIDENT,
PROF. JAMES DEWAR, M.A., LL.D., D.Sc., F.R.S.

THE members of an Association whose studies involve perpetual contemplation of settled law and ordered evolution, whose objects are to seek patiently for the truth of things and to extend the dominion of man over the forces of nature, are even more deeply pledged than other men to loyalty to the Crown and the Constitution which procure for them the essential conditions of calm security and social stability. I am confident that I express the sentiments of all now before me when I say that to our loyal respect for his high office we add a warmer feeling of loyalty and attachment to the person of our Gracious Sovereign. It is the peculiar felicity of the British Association that, since its foundation seventy-one years ago, it has always been easy and natural to cherish both these sentiments, which indeed can never be dissociated without peril. At this, our second meeting held under the present reign, these sentiments are realised all the more vividly, because, in common with the whole empire, we have recently passed through a period of acute apprehension, followed by the uplifting of a national deliverance. The splendid and imposing coronation ceremony which took place just a month ago was rendered doubly impressive both for the King and his people by the universal consciousness that it was also a service of thanksgiving for escape from imminent peril. In offering to His Majesty our most hearty congratulations upon his singularly rapid recovery from a dangerous illness, we rejoice to think that the nation has received gratifying evidence of the vigour of his constitution, and may, with confidence more assured than before, pray that he may have length of happy and prosperous days. No one in his wide dominions is more competent than the King to realise how much he owes, not only to the skill of his surgeons, but also to the equipment which has been placed in their hands as the combined result of scientific investigation in many and diverse directions. He has already displayed a profound and sagacious interest in the discovery of methods for dealing with some of the most intractable maladies that still baffle scientific penetration; nor can we doubt that this interest extends to other forms of scientific investigation, more directly connected with the amelioration of the lot of the healthy than with the relief of the sick. Heredity imposes obligations and also confers aptitude for their discharge. If His Majesty's royal mother throughout her long and beneficent reign set him a splendid example of devotion to the burdensome labours of State which must necessarily absorb the chief part of his energies, his father no less clearly indicated the great part he may play in the encouragement of science. Intelligent appreciation of scientific work and needs is not less but more necessary in the highest quarters to-day than it was forty-three years ago, when His Royal Highness the Prince Consort brought the matter before this Association in the following memorable passage in his Presidential Address:—"We may be justified, however, in hoping that by the gradual diffusion of science and its increasing recognition as a principal part of our national education, the public in general, no less than the legislature and the State, will more and more recognise the claims of science to their

attention; so that it may no longer require the begging box, but speak to the State like a favoured child to its parent, sure of his paternal solicitude for its welfare; that the State will recognise in science one of its elements of strength and prosperity, to protect which the clearest dictates of self-interest demand." Had this advice been seriously taken to heart and acted upon by the rulers of the nation at the time, what splendid results would have accrued to this country! We should not now be painfully groping in the dark after a system of national education. We should not be wasting money, and time more valuable than money, in building imitations of foreign educational superstructures before having put in solid foundations. We should not be hurriedly and distractedly casting about for a system of tactics after confrontation with the disciplined and co-ordinated forces of industry and science led and directed by the rulers of powerful States. Forty-three years ago we should have started fair had the Prince Consort's views prevailed. As it is, we have lost ground which it will tax even this nation's splendid reserves of individual initiative to recover. Although in this country the King rules, but does not govern, the Constitution and the structure of English society assure to him a very potent and far-reaching influence upon those who do govern. It is hardly possible to overrate the benefits that may accrue from his intelligent and continuous interest in the great problem of transforming his people into a scientifically educated nation. From this point of view we may congratulate ourselves that the heir to the Crown, following his family traditions, has already deduced from his own observations in different parts of the empire some very sound and valuable conclusions as to the national needs at the present day.

Griffith—Gilbert—Cornu.

The saddest yet the most sacred duty falling to us on such an occasion as the present is to pay our tribute to the memory of old comrades and fellow-workers whom we shall meet no more. We miss to-day a figure that has been familiar, conspicuous, and always congenial at the meetings of the British Association during the last forty years. Throughout the greater part of that period Mr. George Griffith discharged the onerous and often delicate duties of the assistant general secretary, not only with conscientious thoroughness and great ability, but also with urbanity, tact, and courtesy that endeared him to all. His years sat lightly upon him, and his undiminished alertness and vigour caused his sudden death to come upon us all with a shock of surprise as well as of pain and grief. The British Association owes him a debt of gratitude which must be so fully realised by every regular attender of our meetings that no poor words of mine are needed to quicken your sense of loss, or to add to the poignancy of your regret.

The British Association has to deplore the loss from among us of Sir Joseph Gilbert, a veteran who continued to the end of a long life to pursue his important and beneficent researches with untiring energy. The length of his services in the cause of science cannot be better indicated than by recalling the fact that he was one of the six past Presidents boasting fifty years' membership whose jubilee was celebrated by the Chemical Society in 1898. He was in fact an active member of that Society for over sixty years. Early in his career he devoted himself to a most important but at that time little cultivated field of research. He strove with conspicuous success to place the oldest of industries on a scientific basis, and to submit the complex conditions of agriculture to a systematic analysis. He studied the physiology of plant life in the open air, not with the object of penetrating the secrets of structure, but with the more directly utilitarian aim of establishing the conditions of successful and profitable cultivation. By a long series of experiments alike well conceived and laboriously carried out, he determined the effects of variation in soil, and its chemical treatment—in short, in all the unknown factors with which the

farmer previously had to deal according to empirical and local rules, roughly deduced from undigested experience by uncritical and rudimentary processes of inference. Gilbert had the faith, the insight, and the courage to devote his life to an investigation so difficult, so unpromising, and so unlikely to bring the rich rewards attainable by equal diligence in other directions, as to offer no attraction to the majority of men. The tabulated results of the Rothamsted experiments remain as a benefaction to mankind and a monument of indomitable and disinterested perseverance.

It is impossible for me in this place to offer more than the barest indication of the great place in contemporary science that has been vacated by the lamented death of Professor Alfred Cornu, who so worthily upheld the best traditions of scientific France. He was gifted in a high degree with the intellectual lucidity, the mastery of form, and the perspicuous method which characterised the best exponents of French thought in all departments of study. After a brilliant career as a student, he was chosen at the early age of twenty-six to fill one of the enviable positions more numerous in Paris than in London, the Professorship of Physics at the Ecole Polytechnique. In that post, which he occupied to the end of his life, he found what is probably the ideal combination for a man of science—leisure and material equipment for original research, together with that close and stimulating contact with practical affairs afforded by his duties as teacher in a great school, almost ranking as a department of State. Cornu was admirable alike in the use he made of his opportunities and in his manner of discharging his duties. He was at once a great investigator and a great teacher. I shall not even attempt a summary, which at the best must be very imperfect, of his brilliant achievements in optics, the study of his predilection, in electricity, in acoustics, and in the field of physics generally. As a proof of the great estimation in which he was held, it is sufficient to remind you that he had filled the highest presidential offices in French scientific societies, and that he was a foreign member of our Royal Society and a recipient of its Rumford medal. In this country he had many friends, attracted no less by his personal and social qualities than by his commanding abilities. Some of those here present may remember his appearance a few years ago at the Royal Institution, and more recently his delivery of the Rede Lecture at Cambridge, when the University conferred upon him the honorary degree of Doctor of Science. His death has inflicted a heavy blow upon our generation, upon France, and upon the world.

The Progress of Belfast.

A great man has observed that the "intelligent anticipation of events before they occur" is a factor of some importance in human affairs. One may suppose that intelligent anticipation had something to do with the choice of Belfast as the meeting-place of the British Association this year. Or, if it had not, then it must be admitted that circumstances have conspired, as they occasionally do, to render the actual selection peculiarly felicitous. Belfast has perennial claims, of a kind that cannot easily be surpassed, to be the scene of a great scientific gathering—claims founded upon its scientific traditions and upon the conspicuous energy and success with which its citizens have prosecuted in various directions the application of science to the purposes of life. It is but the other day that the whole nation deplored at the grave of Lord Dufferin the loss of one of the most distinguished and most versatile public servants of the age. That great statesman and near neighbour of Belfast was a typical expression of the qualities and the spirit which have made Belfast what it is, and have enabled Ireland, in spite of all drawbacks, to play a great part in the Empire. I look round on your thriving and progressive city giving evidence of an enormous aggregate of industrial efforts intelligently organised and directed for the building up of a sound social fabric. I find that your great industries are

interlinked and interwoven with the whole economic framework of the Empire, and that you are silently and irresistibly compelled to harmonious co-operation by practical considerations acting upon the whole community. It is here that I look for the real Ireland, the Ireland of the future. We cannot trace with precision the laws that govern the appearance of eminent men, but we may at least learn from history that they do not spring from every soil. They do not appear among decadent races or in ages of retrogression. They are the fine flower of the practical intellect of the nation working studiously and patiently in accordance with the great laws of conduct. In the manifold activities of Belfast we have a splendid manifestation of individual energy working necessarily, even if not altogether consciously, for the national good. In great Irishmen like Lord Dufferin and Lord Roberts, giving their best energies for the defence of the nation by diplomacy or by war, we have complimentary evidence enough to reassure the most timid concerning the real direction of Irish energies and the vital nature of Irish solidarity with the rest of the Empire.

Belfast has played a prominent part in a transaction of a somewhat special and significant kind, which has proved not a little confusing and startling to the easy-going public. The significance of the shipping combination lies in the light it throws on the conditions and tendencies which make such things possible, if not even inevitable. It is an event forcibly illustrating the declaration of his Royal Highness the Prince of Wales, that the nation must "wake up" if it hopes to face its growing responsibilities. Belfast may plead with some justice that it, at least, has never gone to sleep. In various directions an immense advance has been effected during the twenty-eight years that have elapsed since the last visit of the British Association. Belfast has become first a city and then a county, and now ranks as one of the eight largest cities in the United Kingdom. Its municipal area has been considerably extended, and its population has increased by something like 75 per cent. It has not only been extended, but improved and beautified in a manner which very few places can match, and which probably none can surpass. Fine new thoroughfares, adorned with admirable public institutions, have been run through areas once covered with crowded and squalid buildings. Compared with the early fifties, when iron shipbuilding was begun on a very modest scale, the customs collected at the port have increased tenfold. Since the introduction of the power-loom, about 1850, Belfast has distanced all rivals in the linen industry, which continues to flourish notwithstanding the fact that most of the raw material is now imported, instead of being produced, as in former times, in Ulster. Extensive improvements have been carried out in the port at a cost of several millions, and have been fully justified by a very great expansion of trade. These few bare facts suffice to indicate broadly the immense strides taken by Belfast in the last two decades. For an Association that exists for the advancement of science it is stimulating and encouraging to find itself in the midst of a vigorous community, successfully applying knowledge to the ultimate purpose of all human effort, the amelioration of the common lot by an ever-increasing mastery of the powers and resources of Nature.

Tyndall and Evolution.

The Presidential Address delivered by Tyndall in this city twenty-eight years ago will always rank as an epoch-making deliverance. Of all the men of the time, Tyndall was one of the best equipped for the presentation of a vast and complicated scientific subject to the mass of his fellow-men. Gifted with the powers of a many-sided original investigator, he had at the same time devoted much of his time to an earnest study of philosophy, and his literary and oratorical powers, coupled with a fine poetic instinct, were qualifications which placed him in the front rank of the scientific representatives of the later Victorian epoch, and constituted him an exceptionally endowed exponent of scientific thought. In the Belfast dis-

course Tyndall dealt with the changing aspects of the long unsettled horizon of human thought, at last illuminated by the sunrise of the doctrine of evolution. The consummate art with which he marshalled his scientific forces for the purpose of effecting conviction of the general truth of the doctrine has rarely been surpassed. The courage, the lucidity, the grasp of principles, the moral enthusiasm with which he treated his great theme, have powerfully aided in effecting a great intellectual conquest, and the victory assuredly ought to engender no regrets.

Tyndall's views as a strenuous supporter and believer in the theory of evolution were naturally essentially optimistic. He had no sympathy with the lugubrious pessimistic philosophy whose disciples are for ever intent on administering rebuke to scientific workers by reminding them that, however much knowledge man may have acquired, it is as nothing compared with the immensity of his ignorance. That truth is indeed never adequately realised except by the man of science, to whom it is brought home by repeated experience of the fact that his most promising excursions into the unknown are invariably terminated by barriers which, for the time at least, are insurmountable. He who has never made such excursions with patient labour may indeed prattle about the vastness of the unknown, but he does so without real sincerity or intimate conviction. His tacit, if not his avowed, contention is, that since we can never know all, it is not worth while to seek to know more; and that in the profundity of his ignorance he has the right to people the unexplored spaces with the phantoms of his vain imagining. The man of science, on the contrary, finds in the extent of his ignorance a perpetual incentive to further exertion, and in the mysteries that surround him a continual invitation, nay, more, an inexorable mandate. Tyndall's writings abundantly prove that he had faced the great problems of man's existence with that calm intellectual courage, the lack of which goes very far to explain the nervous dogmatism of nescience. Just because he had done this, because he had, as it were, mapped out the boundaries between what is knowable though not yet known and what must remain for ever unknowable to man, he did not hesitate to place implicit reliance on the progress of which man is capable, through the exercise of patient and persistent research. In Tyndall's scheme of thought the chief dicta were the strict division of the world of knowledge from that of emotion, and the lifting of life by throwing overboard the malign residuum of dogmatism, fanaticism, and intolerance, thereby stimulating and nourishing a plastic vigour of intellect. His cry was "Commotion before stagnation, the leap of the torrent before the stillness of the swamp."

His successors have no longer any need to repeat those significant words, "We claim and we shall wrest from theology the entire domain of cosmological theory." The claim has been practically, though often unconsciously, conceded. Tyndall's dictum, "Every system must be plastic to the extent that the growth of knowledge demands," struck a note that was too often absent from the heated discussions of days that now seem so strangely remote. His honourable admissions that, after all that had been achieved by the developmental theory, "the whole process of evolution is the manifestation of a power absolutely inscrutable to the intellect of man," shows how willingly he acknowledged the necessary limits of scientific inquiry. This reservation did not prevent him from expressing the conviction forced upon him by the pressure of intellectual necessity, after exhaustive consideration of the known relations of living things, that matter in itself must be regarded as containing the promise and potency of all terrestrial life. Bacon in his day said very much the same thing: "He that will know the properties and proceedings of matter should comprehend in his understanding the sum of all things, which have been, which are, and which shall be, although no knowledge can extend so far as to singular and individual beings." Tyndall's conclusion was at the time thought to be based on a too

insecure projection into the unknown, and some even regarded such an expansion of the crude properties of matter as totally unwarranted. Yet Tyndall was certainly no materialist in the ordinary acceptance of the term. It is true his arguments, like all arguments, were capable of being distorted, especially when taken out of their context, and the address became in this way an easy prey for hostile criticism. The glowing rhetoric that gave charm to his discourse and the poetic similes that clothed the dry bones of his close-woven logic were attacked by a veritable broadside of critical artillery. At the present day these would be considered as only appropriate artistic embellishments, so great is the unconscious change wrought in our surroundings. It must be remembered that, while Tyndall discussed the evolutionary problem from many points of view, he took up the position of a practical disciple of Nature dealing with the known experimental and observational realities of physical inquiry. Thus he accepted as fundamental concepts the atomic theory, together with the capacity of the atom to be the vehicle or repository of energy, and the grand generalisation of the conservation of energy. Without the former, Tyndall doubted whether it would be possible to frame a theory of the material universe; and as to the latter he recognised its radical significance in that the ultimate philosophical issues therein involved were as yet but dimly seen. That such generalisations are provisionally accepted does not mean that science is not alive to the possibility that what may now be regarded as fundamental may in future be superseded or absorbed by a wider generalisation. It is only the poverty of language and the necessity for compendious expression that oblige the man of science to resort to metaphor and to speak of the Laws of Nature. In reality, he does not pretend to formulate any laws for Nature, since to do so would be to assume a knowledge of the inscrutable cause from which alone such laws could emanate. When he speaks of a "law of Nature," he simply indicates a sequence of events which, so far as his experience goes, is invariable, and which therefore enables him to predict, to a certain extent, what will happen in given circumstances. But, however seemingly bold may be the speculation in which he permits himself to indulge, he does not claim for his best hypothesis more than provisional validity. He does not forget that to-morrow may bring a new experience compelling him to re-cast the hypothesis of to-day. This plasticity of scientific thought, depending upon reverent recognition of the vastness of the unknown, is oddly made a matter of reproach by the very people who harp upon the limitations of human knowledge. Yet the essential condition of progress is that we should generalise to the best of our ability from the experience at command, treat our theory as provisionally true, endeavour to the best of our power to reconcile with it all the new facts we discover, and abandon or modify it when it ceases to afford a coherent explanation of new experience. That procedure is far as are the poles asunder from the presumptuous attempt to travel beyond the study of secondary causes. Any discussion as to whether matter or energy was the true reality would have appeared to Tyndall as a futile metaphysical disputation, which being completely dissociated from verified experience, would lead to nothing. No explanation was attempted by him of the origin of the bodies we call elements, nor how some of such bodies came to be compounded into complex groupings and built up into special structures with which, so far as we know, the phenomena characteristic of life are invariably associated. The evolutionary doctrine leads us to the conclusion that life, such as we know it, has only been possible during a short period of the world's history, and seems equally destined to disappear in the remote future; but it postulates the existence of a material universe endowed with an infinity of powers and properties, the origin of which it does not pretend to account for. The enigma at both ends of the scale Tyndall admitted, and the futility of attempting to answer such

questions he fully recognised. Nevertheless, Tyndall did not mean that the man of science should be debarred from speculating as to the possible nature of the simplest forms of matter or the mode in which life may have originated on this planet. Lord Kelvin, in his Presidential Address, put the position admirably when he said "Science is bound by the everlasting law of honour to face fearlessly every problem that can fairly be presented to it. If a probable solution consistent with the ordinary course of Nature can be found, we must not invoke an abnormal act of Creative Power"; and in illustration he forthwith proceeded to express his conviction that from time immemorial many worlds of life besides our own have existed, and that "it is not an unscientific hypothesis that life originated on this earth through the moss-grown fragments from the ruins of another world." In spite of the great progress made in science, it is curious to notice the occasional recrudescence of metaphysical dogma. For instance, there is a school which does not hesitate to revive ancient mystifications in order to show that matter and energy can be shattered by philosophical arguments, and have no objective reality. Science is at once more humble and more reverent. She confesses her ignorance of the ultimate nature of matter, of the ultimate nature of energy, and still more of the origin and ultimate synthesis of the two. She is content with her patient investigation of secondary causes, and glad to know that since Tyndall spoke in Belfast she has made great additions to the knowledge of general molecular mechanism, and especially of synthetic artifice in the domain of organic chemistry, though the more exhaustive acquaintance gained only forces us the more to acquiesce in acknowledging the inscrutable mystery of matter. Our conception of the power and potency of matter has grown in little more than a quarter of a century to much more imposing dimensions, and the outlook for the future assuredly suggests the increasing acceleration of our rate of progress. For the impetus he gave to scientific work and thought, and for his fine series of researches chiefly directed to what Newton called the more secret and noble works of Nature within the corpuscles, the world owes Tyndall a debt of gratitude. It is well that his memory should be held in perennial respect, especially in the land of his birth.

The Endowment of Education.

These are days of munificent benefactions to science and education, which, however, are greater and more numerous in other countries than in our own. Splendid as they are, it may be doubted, if we take into account the change in the value of money, the enormous increase of population, and the utility of science to the builders of colossal fortunes, whether they bear comparison with the efforts of earlier days. But the habit of endowing science was so long in practical abeyance that every evidence of its resumption is matter for sincere congratulation. Mr. Cecil Rhodes has dedicated a very large sum of money to the advancement of education, though the means he has chosen are perhaps not the most effective. It must be remembered that his aims were political as much as educational. He had the noble and worthy ambition to promote enduring friendship between the great English-speaking communities of the world, and knowing the strength of college ties he conceived that this end might be greatly furthered by bringing together at an English university the men who would presumably have much to do in later life with the influencing of opinion, or even with the direction of policy. It has been held by some a striking tribute to Oxford that a man but little given to academic pursuits or modes of thought should think it a matter of high importance to bring men from our colonies or even from Germany, to submit to the formative influences of that ancient seat of learning. But this is perhaps reading Mr. Rhodes backwards. He shows his affectionate recollection of his college days by his gift to Oriel. But, apart from the main idea of fostering good relations between those who will presumably be influential in England, in the

Colonies, and in the United States, Mr. Rhodes was probably influenced also by the hope that the influx of strangers would help to broaden Oxford notions and to procure revision of conventional arrangements.

Dr. Andrew Carnegie's endowment of Scottish universities, as modified by him in deference to expert advice, is a more direct benefit to the higher education. For while Mr. Rhodes has only enabled young men to get what Oxford has to give, Dr. Carnegie has also enabled his trustees powerfully to augment and improve the teaching equipment of the universities themselves. At the same time he has provided as far as possible for the enduring usefulness of his money. His trustees form a permanent body external to the universities, which, while possessing no power of direct control, must always, as holder of the purse-strings, be in a position to offer independent and weighty criticisms. More recently, Dr. Carnegie has devoted an equal sum of ten million dollars to the foundation of a Carnegie Institution in Washington. Here again he has been guided by the same ideas. He has neither founded a university nor handed over the money to any existing university. He has created a permanent trust charged with the duty of watching educational efforts and helping them from the outside according to the best judgment that can be formed in the circumstances of the moment. Its aims are to be—to promote original research; to discover the exceptional man in every department of study, whether inside or outside of the schools, and to enable him to make his special study his life-work; to increase facilities for higher education; to aid and stimulate the universities and other educational institutions; to assist students who may prefer to study at Washington; and to ensure prompt publication of scientific discoveries. The general purpose of the founder is to secure, if possible, for the United States leadership in the domain of discovery and the utilisation of new forces for the benefit of man. Nothing will more powerfully further this end than attention to the injunction to lay hold of the exceptional man whenever and wherever he may be found, and, having got him, to enable him to carry on the work for which he seems specially designed. That means, I imagine, a scouring of the old world, as well as of the new, for the best men in every department of study—in fact, an assiduous collecting of brains similar to the collecting of rare books and works of art which Americans are now carrying on in so lavish a manner. As in diplomacy and war, so in science, we owe our reputation, and no small part of our prosperity, to exceptional men; and that we do not enjoy these things in fuller measure we owe to our lack of an army of well-trained ordinary men capable of utilising their ideas. Our exceptional men have too often worked in obscurity, without recognition from a public too imperfectly instructed to guess at their greatness, without assistance from a State governed largely by dilettantes, and without help from academic authorities hidebound by the pedantries of medieval scholasticism. For such men we have to wait upon the will of Heaven. Even Dr. Carnegie will not always find them when they are wanted. But what can be done in that direction will be done by institutions like Dr. Carnegie's, and for the benefit of the nation that possesses them in greatest abundance and uses them most intelligently. When contemplating these splendid endowments of learning, it occurred to me that it would be interesting to find out exactly what some definite quantity of scientific achievements has cost in hard cash. In an article by Carl Snyder in the January number of the *North American Review*, entitled, "America's Inferior Place in the Scientific World," I found the statement that "it would be hardly too much to say that during the hundred years of its existence the Royal Institution alone has done more for English science than all of the English universities put together. This is certainly true with regard to British industry, for it was here that the discoveries of Faraday were made." I was emboldened by this estimate from a distant and impartial observer to

do, what otherwise I might have shrunk from doing, and to take the Royal Institution—after all, the foundation of an American citizen, Count Rumford—as the basis of my inquiry. The work done at the Royal Institution during the past hundred years is a fairly definite quantity in the mind of every man really conversant with scientific affairs. I have obtained from the books accurate statistics of the total expenditure on experimental inquiry and public demonstrations for the whole of the nineteenth century. The items are:—

Professors' salaries—Physics and Chemistry ..	£54,600
Laboratory expenditure	24,430
Assistants' salaries	21,590

Total for one hundred years £100,620

In addition, the members and friends of the Institution have contributed to a fund for exceptional expenditure for Experimental Research the sum of £9580. It should also be mentioned that a Civil List pension of £300 was granted to Faraday in 1853, and was continued during twenty-seven years of active work and five years of retirement. Thirty-two years in all, at £300 a year, make a sum of £9600, representing the national donation, which, added to the amount of expenditure just stated, brings up the total cost of a century of scientific work in the laboratories of the Royal Institution, together with public demonstrations, to £119,800, or an average of £1200 per annum. I think if you recall the names and achievements of Young, Davy, Faraday, and Tyndall, you will come to the conclusion that the exceptional man is about the cheapest of natural products. It is a popular fallacy that the Royal Institution is handsomely endowed. On the contrary, it has often been in financial straits; and since its foundation by Count Rumford its only considerable bequests have been one from Thomas G. Hodgkins, an American citizen, for Experimental Research, and that of John Fuller for endowing with £95 a year the chairs of Chemistry and Physiology. In this connection the Davy-Faraday Laboratory, founded by the liberality of Dr. Ludwig Mond, will naturally occur to many minds. But though affiliated to the Royal Institution, with, I hope, reciprocal indirect advantages, that Laboratory is financially independent and its endowments are devoted to its own special purpose, which is to provide opportunity to prosecute independent research for worthy and approved applicants of all nationalities. The main reliance of the Royal Institution has always been, and still remains, upon the contributions of its members, and upon corresponding sacrifices in the form of time and labour by its professors. It may be doubted whether we can reasonably count upon a succession of scientific men able and willing to make sacrifices which the conditions of modern life tend to render increasingly burdensome. Modern science is in fact in something of a dilemma. Devotion to abstract research upon small means is becoming always harder to maintain, while at the same time the number of wealthy independent searchers after truth, and patrons of science of the style of Joule, Spottiswoode, and De la Rue, is apparently becoming smaller. The installations required by the refinements of modern science are continually becoming more costly, so that upon all grounds it would appear that without endowments of the kind provided by Dr. Carnegie the outlook for disinterested research is rather dark. On the other hand, these endowments, unless carefully administered, might obviously tend to impair the single-minded devotion to the search after truth for its own sake, to which science has owed almost every memorable advance made in the past. The Carnegie Institute will dispose in a year of as much money as the members of the Royal Institution have expended in a century upon its purely scientific work. It will at least be interesting to note how far the output of high-class scientific work corresponds to the hundredfold application of money to its production. Nor will it be of less interest to the people of this country to observe the results obtained from that

moiety of Dr. Carnegie's gift to Scotland which is to be applied to the promotion of scientific research.

Applied Chemistry, English and Foreign.

The Diplomatic and Consular reports published from time to time by the Foreign Office are usually too belated to be of much use to business men, but they sometimes contain information concerning what is done in foreign countries which affords food for reflection. One of these reports, issued a year ago, gives a very good account of the German arrangements and provisions for scientific training, and of the enormous commercial demand for the services of men who have passed successfully through the Universities and Technical High Schools, as well as of the wealth that has accrued to Germany through the systematic application of scientific proficiency to the ordinary business of life.

Taking these points in their order, I have thought it a matter of great interest to obtain a comparative view of chemical equipment in this country and in Germany, and I am indebted to Professor Henderson of Glasgow, who last year became the secretary of a committee of this Association of which Professor Armstrong is chairman, for statistics referring to this country, which enable a comparison to be broadly made. The author of the consular report estimates that in 1901 there were 4500 trained chemists employed in German works, the number having risen to this point from 1700 employed twenty-five years earlier. It is difficult to give perfectly accurate figures for this country, but a liberal estimate places the number of works chemists at 1500, while at the very outside it cannot be put higher than somewhere between 1500 and 2000. In other words, we cannot show in the United Kingdom, notwithstanding the immense range of the chemical industries in which we once stood prominent, more than one-third of the professional staff employed in Germany. It may perhaps be thought or hoped that we make up in quality for our defect in quantity, but unfortunately this is not the case. On the contrary, the German chemists are, on the average, as superior in technical training and acquirements as they are numerically. Details are given in the report of the training of 633 chemists employed in German works. Of these, 69 per cent hold the degree of Ph.D., about 10 per cent hold the diploma of a Technical High School, and about 5 per cent hold both qualifications. That is to say, 84 per cent have received a thoroughly systematic and complete chemical training, and 74 per cent of these add the advantages of a university career. Compare with this the information furnished by 500 chemists in British works. Of these, only 21 per cent are graduates, while about 10 per cent hold the diploma of a college. Putting the case as high as we can, and ignoring the more practical and thorough training of the German universities, which give their degrees for work done, and not for questions asked and answered on paper, we have only 31 per cent of systematically trained chemists against 84 per cent in German works. It ought to be mentioned that about 21 per cent of the 500 are Fellows or Associates of the Institute of Chemistry, whatever that may amount to in practice, but of these a very large number have already been accounted for under the heads of graduates and holders of diplomas. These figures, which I suspect are much too favourable on the British side, unmistakably point to the prevalence among employers in this country of the antiquated adherence to rule of thumb, which is at the root of much of the backwardness we have to deplore. It hardly needs to be pointed out to such an audience as the present that chemists who are neither graduates of a university nor holders of a diploma from a technical college, may be competent to carry on existing processes according to traditional methods, but are very unlikely to effect substantial improvements, or to invent new and more efficient processes. I am very far from denying that here and there an individual may be found whose exceptional ability enables him to triumph over all defects of training. But in all educational matters it is the average

man whom we have to consider, and the average ability which we have to develop. Now, to take the second point—the actual money value of the industries carried on in Germany by an army of workers both quantitatively and qualitatively so superior to our own. The Consular report estimates the whole value of German chemical industries at not less than fifty millions sterling per annum. These industries have sprung up within the last seventy years, and have received enormous expansion during the last thirty. They are, moreover, very largely founded upon basic discoveries made by English chemists, but never properly appreciated or scientifically developed in the land of their birth. I will place before you some figures showing the growth of a single firm engaged in a single one of these industries—the utilisation of coal-tar for the production of drugs, perfumes, and colouring-matters of every conceivable shade. The firm of Friedrich Bayer and Co. employed in 1875, 119 workmen. The number has more than doubled itself every five years, and in May of this year that firm employed 5000 workmen, 160 chemists, 260 engineers and mechanics, and 680 clerks. For many years past it has regularly paid 18 per cent on the ordinary shares, which this year has risen to 20 per cent; and in addition, in common with other and even larger concerns in the same industry, has paid out of profits for immense extensions usually charged to capital account. There is one of these factories the works and plant of which stand in the books at £1,500,000, while the money actually sunk in them approaches to £5,000,000. In other words, the practical monopoly enjoyed by the German manufacturers enables them to exact huge profits from the rest of the world, and to establish a position which, financially as well as scientifically, is almost unassailable. I must repeat that the fundamental discoveries upon which this gigantic industry is built were made in this country, and were practically developed to a certain extent by their authors. But in spite of the abundance and cheapness of the raw material, and in spite of the evidence that it could be most remuneratively worked up, these men founded no school and had practically no successors. The colours they made were driven out of the field by newer and better colours made from their stuff by the development of their ideas, but these improved colours were made in Germany and not in England. Now what is the explanation of this extraordinary and disastrous phenomenon? I give it in a word—want of education. We had the material in abundance when other nations had comparatively little. We had the capital and we had the brains, for we originated the whole thing. But we did not possess the diffused education without which the ideas of men of genius cannot fructify beyond the limited scope of an individual. I am aware that our patent laws are sometimes held responsible. Well, they are a contributory cause; but it must be remembered that other nations with patent laws as protective as could be desired have not developed the colour industry. The patent laws have only contributed in a secondary degree, and if the patent laws have been bad the reason for their badness is again want of education. Make them as bad as you choose, and you only prove that the men who made them, and the public whom these men try to please, were misled by theories instead of being conversant with fact and logic. But the root of the mischief is not in the patent laws or in any legislation whatever. It is in the want of education among our so-called educated classes, and secondarily among the workmen on whom these depend. It is in the abundance of men of ordinary plodding ability, thoroughly trained and methodically directed, that Germany at present has so commanding an advantage. It is the failure of our schools to turn out, and of our manufacturers to demand, men of this kind, which explains our loss of some valuable industries and our precarious hold upon others. Let no one imagine for a moment that this deficiency can be remedied by any amount of that technical training which is now the fashionable nostrum. It is an excellent thing,

no doubt, but it must rest upon a foundation of general training. Mental habits are formed for good or evil long before men go to the technical schools. We have to begin at the beginning: we have to train the population from the first to think correctly and logically, to deal at first hand with facts, and to evolve, each one for himself, the solution of a problem put before him, instead of learning by rote the solution given by somebody else. There are plenty of chemists turned out, even by our Universities, who would be of no use to Bayer and Co. They are chockfull of formulæ, they can recite theories, and they know text-books by heart; but put them to solve a new problem, freshly arisen in the laboratory, and you will find that their learning is all dead. It has not become a vital part of their mental equipment, and they are floored by the first emergence of the unexpected. The men who escape this mental barrenness are men who were somehow or other taught to think long before they went to the university. To my mind, the really appalling thing is not that the Germans have seized this or the other industry, or even that they may have seized upon a dozen industries. It is that the German population has reached a point of general training and specialised equipment which it will take us two generations of hard and intelligently directed educational work to attain. It is that Germany possesses a national weapon of precision which must give her an enormous initial advantage in any and every contest depending upon disciplined and methodised intellect.

(To be continued).

THE RADIO-ACTIVITY OF THORIUM COMPOUNDS.*

II. THE CAUSE AND NATURE OF RADIO-ACTIVITY.

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and
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(Continued from p. 101).

7. The Place of Emanating Power in the Radio-activity of Thorium.

TURNING from radio-active solids to gases which manifest this property, a great many of the very puzzling results obtained in the investigation of the radio-active emanation produced by thorium compounds can now also be simply explained. This section is devoted to this purpose and to further work that has been done in order to more exactly determine the place of emanating power in the radio-activity of thorium.

It was shown that the solutions from which thorium hydroxide had been precipitated by ammonia possessed about as much emanating power as the solutions from which they were prepared, while the precipitated hydroxide in all cases but one were more or less completely de-emanated, but spontaneously regained their normal value with lapse of time. It will be recalled that in one solitary instance that could not be repeated (*Trans. Chem. Soc.*, 1902, p. 344; *CHEMICAL NEWS*, vol. lxxv., p. 306) the hydroxide possessed an abnormally high emanating power, decaying to nearly normal value in fourteen days. This can now be explained. The thorium was first partly precipitated as thorium carbonate by means of sodium carbonate, and nitric acid added to re-dissolve a part. Under these circumstances all the ThX remains in solution, and on adding ammonia the thorium only should be precipitated, which, as always occurred when the experiment was repeated, ought to be more or less free from emanating power. But if as apparently happened in the experimental case, care was not taken to boil off all the carbon dioxide produced before adding ammonia, the latter would re-form a carbonate which completely pre-

* Communicated by the Authors.

precipitates both ThX and thorium. Hence the small hydroxide fraction contained all the ThX originally present, and possessed a high emanating power decaying to normal value with time. The fact that the carbonate fraction in this same experiment behaved abnormally, and did not recover its emanating power, is more difficult to explain, and the point will be reverted to later.

In further explanation of the results then obtained, it is only necessary to point out that since emanating power and not radio-activity was the first object of the investigation, any measurements of the latter, especially in the earlier part of the paper, were, as a rule, performed long after the specimens had been prepared; i.e., after they had regained their normal values. In some cases if these measurements had been made immediately after preparation the results would have doubtless been different.

The conclusion was arrived at that emanating power was the manifestation of a dynamical change of the nature of a chemical reaction, rather than a function of matter in the static state, even before it was known that the emanating material, ThX, was being continuously reproduced. The latter discovery, however, enables a fairly complete explanation of the phenomenon to be given.

On resuming the work after the Christmas vacation, it was found that, on the one hand, some concentrated filtrates possessing high emanating power originally had completely lost it with lapse of time. On the other hand, the emanating power of almost all of the numerous samples of thorium hydroxide and carbonate prepared had recovered to about the same value, viz., between three and four times that of thorium oxide.

The rate of decay of the emanating power of ThX, and the recovery of this property by the thorium from which it had been separated, were then investigated in parallel with the similar experiments on radio-activity already described. One quarter of the concentrated filtrate used for the latter purpose was taken, and the decrease of its emanating power with time measured. The increase of emanating power of the thorium hydroxide from which it had been prepared was also measured. The curves (Fig. 3) express the results. The decay curve is merely approximate; for it is not easy to accurately take the emanating power of a liquid without special arrangements to ensure the constancy of the air current and the shaking of the solution. The experiments, although merely approximate, bear out the conclusion that emanating power decays and recovers according to the same law as the radio-activity of ThX, and that it is therefore one of the properties of the latter and not of thorium. If care be taken to remove the ThX, the thorium almost entirely loses its emanating power. The small fraction that remains—often only a few per cent of the maximum—can be accounted for by the reproduction of ThX during the time taken to dry the precipitate. The decay curve given, so far as it can be relied on, shows that the emanating power of ThX at any instant is proportional to its radio-activity.

It was shown in the first paper that the emanation consisted of a chemically inert gas continuously emitted from thorium compounds. The results, therefore, find their simplest expression on the view that, just as a chemical change is proceeding in thorium whereby a non-thorium material is produced, so the latter undergoes a further reaction, giving rise to a gaseous product which in the radio-active state constitutes the emanation.

It will be seen at once that this secondary change is of a different kind from the primary, for it is affected apparently by the conditions in a very marked manner. It was shown that moisture, the state of aggregation and temperature, influenced the value of emanating power. From -80° to a red-heat the latter regularly increases in the ratio of 1:40 in the case of thorium oxide, while the ratio between the values for thorium nitrate in the solid state and in solution is as 1:200. The secondary reaction appears, therefore, at first sight much more nearly allied to ordinary chemical reaction than the primary. It must

not be forgotten, however, that the laws controlling the manifestation of the two phenomena—radio-activity and emanating power—are of necessity very different. In the former we deal with the intensity of radiations emitted by a solid, in the latter with the rate of escape of a gas into the surrounding air from either a solid or a liquid. Since this gas is detected by its radio-activity, and this decays extremely rapidly with time, a very slight delay in the rate of its escape will enormously affect the experimental value obtained for emanating power. It is possible that this cause is sufficient to account for the results obtained at different temperatures and with solids and liquids. De-emanation by ignition on this view would mean that at a certain temperature the crystalline form of thorium is permanently altered in such a way that the emanation is delayed in its escape.

On the other hand, it is now well established by experiment that sometimes thorium compounds de-emanated chemically by removal of ThX do not recover their normal emanating power with time, but remain constant at a lower value. The carbonate mentioned in the last paper, and already referred to, is an example of this; for it possessed hardly any emanating power until it was again dissolved and precipitated. On one occasion two samples of hydroxide prepared from different nitrates were tested together for rise of emanating power. That of the one rose normally to its maximum (as in Fig. 3), which was twenty times the minimum. The other started from the same minimum, but rose to a maximum only one-fourth as great. When the experiment was repeated under the same conditions, using the same sample of nitrate, the compound behaved normally. It thus appears that the emanation can be almost entirely prevented from escaping in the radio-active state in some cases, and partially prevented in others, where no visible peculiarity of physical condition exists, and where other preparations similarly prepared behave normally.

The question is further complicated by the property possessed by the emanation of exciting radio-activity on all surfaces with which it comes into contact. This process must be going on in the matter of the thorium compound itself, and it will be shown (Section 8) that this effect contributes an important quota to the total radio-activity of the compound. It seemed reasonable to suppose that the effect will be the greater, the less emanation succeeds in escaping in the radio-active state, and therefore that de-emanated compounds should possess a greater proportion of excited radio-activity than those with high emanating power. This conclusion was tested by converting a specimen of thorium carbonate with an emanating power five times that of ordinary thorium into oxide, and de-emanating by intense ignition. The energy that before escaped in the form of emanation is now all but a few per cent prevented from escaping. The radio-activity of the oxide so prepared rose in the first three days about 30 per cent of its original amount, and there thus seem to be grounds for the view that the excited radio-activity will contribute a much greater effect in a non-emanating thorium compound than in one possessing great emanating power. Additional confirmation of this view is to be found in the nature of the radiations emitted by the two classes of compounds (Section 10).

It will be seen that the phenomenon is too complicated to allow of an answer at the present stage to the question whether the secondary change which produces the emanation is, like the primary, independent of the conditions or not.

8. The Initial Portions of the Curves of Decay and Recovery.

The curves of the recovery and decay of the activities of thorium and ThX with time suggested the explanation that the radio-activity of thorium was being maintained by the production of ThX at a constant rate. Before this can be considered rigidly established two outstanding points remain to be cleared up:—1. What is the meaning

of the early portion of the curves? The recovery curve drops before it rises, and the decay curve rises before it drops. 2. Why does not the removal of ThX render thorium completely inactive? A large proportion of the original radio-activity remains after the removal of ThX.

A study of the curves (Fig. 1) shows that in each case a double action is probably at work. It must be supposed that the normal decay and recovery are taking place, but are being masked by a simultaneous rise and decay from causes unknown. From what is known of thorium radio-activity it was surmised that an action might be taking place similar to that effected by the emanation of exciting radio-activity on surrounding inactive matter. On this view the residual activity of thorium might consist in whole or in part of a secondary or excited radio-activity produced on the whole mass of the thorium compound by its association with the ThX. The drop in the recovery curve on this view would be due to the decay of this excited radio-activity proceeding simultaneously with and at first reversing the effect of the regeneration of ThX. The rise of the decay curve would be the increase due to the ThX exciting activity on the matter with which it is associated, the increase from this cause being greater than the decrease due to the decay of the activity of the ThX. It is easy to put this hypothesis to experimental test. If the ThX is removed from the thorium as soon as it is formed over a sufficient period, the former will be prevented from exciting activity on the latter, and that already excited will decay spontaneously. The experiment was therefore performed. A quantity of nitrate was precipitated as hydroxide in the usual way to remove ThX, the precipitate re-dissolved in nitric acid, and again precipitated after a certain interval. From time to time a portion of the hydroxide was removed, and its radio-activity tested. In this way the thorium was precipitated in all twenty-three times in a period of nine days, and the radio-activity reduced to a constant minimum. The following table shows the results:—

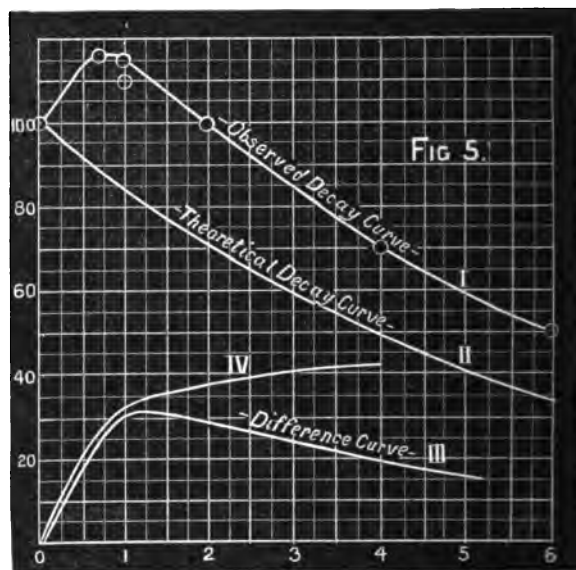
After precipitations—	Activity of hydroxide.
At three intervals of twenty-four hours	39 per cent
At three more intervals each of twenty-four hours, and three more each of eight hours.. . . .	22 per cent
At three more each of eight hours ..	24 per cent
At six more each of four hours ..	25 per cent

The constant minimum thus attained of about 25 per cent the original activity is thus about 21 per cent below that obtained by two successive precipitations without interval, which has been shown to remove all the ThX separable by the process. The rate of recovery of this twenty-three times precipitated hydroxide was then measured (Fig. 4). It will be seen that it is now quite normal, and the initial drop characteristic of the ordinary curve is quite absent. It is, in fact, almost identical with the ordinary curve (Fig. 1) that has been produced back to cut the vertical axis, and there is thus no doubt that there is a residual activity of thorium unconnected apparently with ThX, and constituting about one-fourth of the whole.

The decay curves of several of the fractions of ThX separated in this experiment after varying intervals of time were taken for the first few days. All of them showed the initial rise of about 15 per cent at the end of eighteen hours, and then a normal decay to zero. The position is thus proved that the initial irregularities are caused by the secondary radiation excited by ThX upon the surrounding matter. By suitably choosing the conditions the recovery curve can be made to rise normally from a constant minimum, and the decay curve be shown to consist of two curves—the first the rate of production of excited radio-activity, and the second the rate of decay of the activity as a whole. It is a significant fact that exactly similar curves have already been obtained by one of us (*Phys. Zeit.*, 1900, p. 254) for the excited radio-activity produced by the thorium emanation under very similar conditions. If a negatively charged wire be ex-

posed for a few minutes only to the thorium emanation, the excited radio-activity produced at first increases to several times its value for the first few hours after the exciting cause is removed, and then commences to decay, exactly as in the case of ThX.

So far nothing has been stated as to whether the excited radio-activity, which contributes about 21 per cent of the total activity of thorium, is the same or different from the known type produced by the thorium emanation. All that has been assumed is that it should follow the same general law, i.e., the effect will increase with the time of action of the exciting cause, and decrease with time after the cause is removed. If the rate of rise of the excited activity be worked out from the curves given (Fig. 5), it will



be found to agree with that of the ordinary excited activity, i.e., it rises to half value in about twelve hours. Curve 1 is the observed decay curve for ThX. Curve 2 is the theoretical curve, assuming that it decreases geometrically with the time, and falls to half value in four days. Curve 3 is obtained by plotting the difference between these two, and therefore constitutes the curve of excited activity. Curve 4 is the experimental curve obtained for the rise of the excited radio-activity from the thorium emanation when the exciting cause is constant. But the exciting cause (ThX) in the present case is not constant, but is itself falling to half value in four days, and hence the difference curve, at first almost on the other, drops away from it as time goes on, and finally decays to zero. Curve 3, Fig. 1, is a similar difference curve, representing the decay of excited activity, plotted from the recovery curves of thorium. There is thus no reason to doubt that the effect is the same as that produced by the thorium emanation, which is itself a secondary effect of ThX.

9. The Non-separable Radio-activity of Thorium.

It has not yet been found possible by any means to free thorium from its residual activity, and the place of this part in the scheme of radio-activity of thorium remains to be considered. Disregarding the view that it is a separate phenomenon and not connected with the major part of the activity, two hypotheses can be brought forward capable of experimental test, and, in accordance with the views advanced on the nature of radio-activity, to account for existence of this part. First, if there was a second type of excited activity produced by ThX, similar to that known, but with a very slow rate of decay, it would account for the existence of the non-separable activity. If this is true

it will not be found possible to free thorium from this activity by chemical means, but the continuous removal of ThX over a very long period would, as in the above case, cause its spontaneous decay.

Secondly, if the change which gives rise to ThX produces a second type of matter at the same time, *i.e.*, if it is of the type of a decomposition rather than a depolymerisation, the second type would also in all probability be radio-active, and would cause the residual activity. On this view the second type of matter should also be amenable to separation by chemical means, although it is certain from the failure of the methods already tried that it resembles thorium much more closely than ThX. But until it is separated from the thorium producing it, its activity will not decay spontaneously. Thus, what has already been shown to hold for ThX will be true for the second constituent if methods are found to remove it from the thorium.

It will be shown in a following communication by one of us that uranium also possesses a non-separable radio-activity extremely analogous to that possessed by thorium, and whatever view is taken of the one will in all probability hold also for the other. This consideration makes the second hypothesis, that the residual activity is caused by a second non thorium type of matter produced in the original change, the more probable of the two.

(To be continued).

NOTE ON THE DETECTION OF ARSENIC AND SELENIUM IN SULPHUR.

By FRED. W. STEEL, F.C.S.

HAVING tried a number of methods* for the detection of arsenic and selenium in sulphur without getting satisfactory results, I devised the following one, by use of which very minute quantities of these elements can easily and with certainty be detected.

So far as I can learn from the literature at my disposal, which is fairly voluminous, the method detailed below has not been previously published. The procedure is as follows:—

Two hundred grms. of the finely-ground sample are placed in a cylinder of filter-paper in a 200 c.c. Soxhlet fat extractor, and extracted in the usual way by freshly-distilled pure carbon bisulphide. I allow about four hours—or longer—for this operation. When extraction is complete, the paper cylinder is withdrawn from the Soxhlet, and the carbon bisulphide with which it is soaked allowed to evaporate. The residue on the paper is then scraped into a No. 3 beaker, 50 c.c. strong nitric acid added, and the whole heated on the sand-bath (or, preferably, on the water-bath, if the results are not required immediately) until all nitric acid is driven off. One c.c. strong hydrochloric acid is next added; then, after a few minutes, about 5 c.c. warm water. The contents of beaker are well stirred, filtered through a small filter into a 7" x 1" test-tube, and the filter washed twice with the least possible quantity of water.

— Now add to the contents of the test-tube 20 c.c. strong hydrochloric acid, 1 or 2 c.c. of a fairly concentrated solution of stannous chloride in hydrochloric acid, and, lastly, 5 c.c. strong sulphuric acid. Warm gently, cover test-tube with a watch-glass, and allow to stand for some hours (preferably over night).

If arsenic—or selenium insoluble in carbon bisulphide (which I often find)—is present in the sulphur under examination, a dark brown precipitate settles down. The solution is filtered through a layer of asbestos (purified by boiling with aqua regia, washing, and igniting) on a

platinum filtering cone in an ordinary glass funnel, using the suction-pump.

The precipitate and asbestos are washed with cold water until perfectly free from acid, then removed from the platinum cone and dried at 100° C. When quite dry, they are transferred to a hard glass tube (about 7 m.m. bore) closed at one end; the tube is then drawn out, immediately above its contents, to about 1·5 m.m.; the narrow part being left about 80 m.m. long. The bulb portion, containing the asbestos, &c., is now heated to redness in a Bunsen flame. Arsenic and selenium, if present, are driven off and condense in the narrow part of the tube; the selenium generally appears in both the red and black modifications, and is at once identified by the characteristic odour which may be perceived at the open end of the tube, and which is an extremely delicate and unmistakable indication of its presence.

The part of the tube containing the sublimate is cut off and placed in a fresh ignition tube, similar to the first; this tube is drawn out in exactly the same manner, leaving a fair sized bulb, so that the arsenic and selenium may be oxidised as completely as possible on heating.*

The bulb of the ignition tube is now gradually heated, holding the tube horizontally; the arsenic and selenium are oxidised and volatilised, the oxides and some unoxidised selenium condensing in the narrow part of the tube.

The arsenious oxide forms a ring of beautiful glistening crystals, which can be identified with ease with a moderate microscopic power.

The selenium dioxide—in the entire absence of water—forms fern-shaped crystals. There is, however, nearly always a trace of water present, and therefore a solution of the oxide in minute globules is generally found in place of the crystals. In any case, it cannot be confounded with the arsenious oxide, the difference being very marked. The narrow part of the tube, containing the sublimate, is now sealed off, labelled, and kept for reference.

It is, of course, necessary to perform a blank test to ensure that the sulphuric, hydrochloric, and nitric acids used do not contain arsenic or selenium. In the case of the stannous chloride any precipitate which forms when the solution is prepared must be removed; the solution is then ready for use at any time.

If selenium has not been found associated with the arsenic, it will be necessary to distil off the carbon bisulphide from the extracted sulphur in the extraction flask; carefully break up the mass of sulphur, remove it from the flask, dry, and powder finely; return it to the flask, add about 200 c.c. strong (preferably pure, fuming) nitric acid, and digest in the water-bath for six hours, keeping a small funnel in flask neck to retard evaporation of nitric acid. The contents of the flask are now filtered through asbestos, the unoxidised sulphur washed twice with water, the filtrate then evaporated until copious white fumes of sulphuric acid appear; solution is poured (after cooling) into a 7" x 1" test-tube, 20 c.c. strong hydrochloric acid, then 1 or 2 c.c. of the stannous chloride solution mentioned above are added. Selenium, if present, is precipitated after standing, and may be identified by formation of the red sublimate in ignition tube, and also by odour on igniting.

Experiments are being undertaken to ascertain if the above method can be applied quantitatively.

Chemical Laboratory,
Cumings, Smith, and Co., Pty., Ltd.,
Yarraville, Melbourne, July 29, 1902.

Electro-chemical Equivalent of Silver.—A. Leduc.—

The mass of silver deposited on the cathode by one coulomb usually depends on several circumstances. It seems, however, that it is possible to obtain a result accurate to one ten-thousandth in the determination of the electro-chemical equivalent of this metal by operating with a perfectly neutral or even basic bath at the beginning of the experiment, and so avoiding the formation of acid at the anode.—*Comptes Rendus*, cxxxv., No. 4.

* Arsenic appears to be more readily oxidised than selenium.

* In particular I may mention that of Schaeppi (*vide Crookes's "Select Methods in Chemical Analysis,"* Third Edition, p. 423), by which I have been quite unable to get anything approaching reliable results, owing to the fact that very dilute ammonium hydrate dissolves sulphur just as readily as it does arsenious sulphide.

OBITUARY.

SIR FREDERICK ABEL.

We regret to announce the death of the distinguished chemist, Sir Frederick Abel, which occurred suddenly on Saturday night at his residence in Whitehall Court.

Frederick Augustus Abel was born in London on July 17, 1827, being the eldest son of J. L. Abel, of Woolwich, and grandson of A. C. A. Abel, Court miniature painter to the Grand Duke of Mecklenburg-Schwerin. Undeterred by warnings that it offered very slight prospects as a career, he determined to adopt chemistry as his profession, and was at once met by the difficulties which confronted every one of limited means, who, in the early years of Queen Victoria's reign, desired good scientific instruction at moderate expense. The Royal Polytechnic Institution was practically the only available place where the latter requirement was fulfilled, and, accordingly, in 1844, Abel's name was entered as a pupil on its books. But it was not long before he found that no attempt was made at methodical instruction; indeed, he soon began to doubt whether the "professor" had the ability to teach, even if he had had the wish. After spending six months in working through a standard textbook of chemistry according to his own devices, he decided that he had had enough of the Polytechnic Institution, and took his leave, the proud possessor of a testimonial setting forth the skill and minuteness with which his analyses had been conducted, and confidently recommending him to any post where a knowledge of practical chemistry might be required. But, luckily for him, a scheme for establishing in London a school of chemistry on the lines of Liebig's famous one at Giessen was successfully carried through about this time, and thus he was able to form one of the twenty-six students on the roll of the Royal College of Chemistry, when its temporary laboratory in George Street, Hanover Square, was opened in the autumn of 1845, under the direction of the famous German chemist, A. W. von Hofmann. At the college he spent some six years, during five of which he was one of Hofmann's assistants, and he only left to succeed Faraday as Professor of Chemistry at the Royal Military Academy. A few years later, in 1854, he was appointed Chemist to the War Office—a position which he filled for thirty-four years. His official duties naturally led him to devote attention to the study of explosives for military purposes, and thus he became intimately concerned in some of the most important developments that the latter part of last century witnessed in the manufacture and use of ammunition. Perhaps the most striking of these was the supersession of black powder by the so-called smokeless powders, and it may almost be said that Abel rendered this change possible. Of most of these powders, at any rate of the most successful ones, gun-cotton is a chief constituent. Now, though he was not the discoverer of gun-cotton, nor even the first to investigate its properties, he devised such improvements in the processes of preparation as to enable it to be safely manipulated and efficiently applied to practical uses. In Austria Baron von Lenk had endeavoured to produce the substance in a form which could be utilised in firearms, but his system of regulating the rapidity of burning by special arrangements of the threads achieved but indifferent success. In 1862 Austria communicated to the British Government the details of von Lenk's work, on the basis of which Abel was instructed, as the War Office chemist, to carry out experiments on the substance and inquire into its manufacture, constitution, stability, &c. His researches resulted in the elaboration of the system of preparation which has come into extensive use not only in this country but also in Germany, France, and elsewhere. After being nitrated, the cotton is reduced to pulp, as in the process of making paper. In this condition it can be more thoroughly washed than when in a fibrous

state, and thus can be completely freed from all traces of acid, the presence of which is so dangerous to its stability. When purified, the pulp is compressed by hydraulic power into homogeneous masses of any desired shape or size, and in this way control is gained over the rapidity of explosion, which is greatly modified according to the mechanical condition of the material. It is claimed that, when all the stipulated regulations are strictly observed, the preparation of gun-cotton, though it is a far stronger explosive, is much safer than that of ordinary black powder.

The possibility of utilising this substance for the production of smokeless powders had occurred to Abel, as to others, long before they became fashionable, but it was not until reports began to arrive of the wonderful results that were being attained with them by Continental nations that he received any official encouragement to pursue the subject. In 1888 the British Government had become alive to the situation, and appointed a special committee to examine the various kinds of smokeless powder then in existence, and to report which of them was best adapted to the requirements of the British Army and Navy. The problem was complicated by the fact that it was not so much the best powder absolutely that had to be selected as the powder best suited to the peculiarities of the Lee-Metford rifle. That weapon in its then form had an extraordinarily rapid twist in its rifling, and the difficulty was to get a powder such as would, with the charge available in the chamber, be able to overcome the resistance of the rifling and yet project the bullet with the desired muzzle velocity. The committee, of which Abel was president, spent some years in their inquiry, investigating every powder of which they could obtain samples and testing the properties of each by minute experiments. In the end they failed to find anyone the adoption of which they could unreservedly recommend. Compounds were brought to their notice which, with certain modifications, might have proved suitable, but, as their inventors declined to make the required changes, the idea of adopting any of them had to be abandoned. In these circumstances the committee set themselves to devise a powder which should be as free as possible from the defects noticed in the mixtures submitted to examination, and the result of their labours was that Sir Frederick Abel, in conjunction with Professor Dewar, patented the substance which is now the standard powder of the British services, and which is known as cordite, in allusion to the cord-like form in which it is prepared. All things considered, cordite, of which the main constituents are gun-cotton and nitro-glycerin, may, perhaps, be described as the best smokeless powder in existence, and it would probably be still better at the present time if those responsible for its manufacture had paid more attention to suggestions made for its improvement by its original inventors; at any rate, that appreciation of its merits which is implied by extensive imitation has been accorded it abroad, while at home several inventors have paid it the compliment of attempting to prove it to be the precise material which their patent specifications were designed to cover.

In addition to his work on gun-cotton, Sir Frederick Abel, in conjunction with Sir Andrew Noble, carried out what is probably the most complete investigation into the processes attendant on the firing of black powder that has ever been attempted. Charges up to 20 lbs. were exploded in perfectly closed vessels, and the resulting products submitted to analysis. In this way the enormous influence of the size of the grains and of the pressure under which they were fired was made apparent. The experiments showed that any attempt to construct a general chemical expression for the metamorphosis of normal powder was of no practical value whatever, because the same powder fired under different pressures, and the same powder fired at the same pressure, but in different sized grains, gave quite different products. To the theory of detonation Abel made important contributions, his ingenious experiments throwing much light on the conditions of its occurrence

and the rapidity of its transmission. The latter, he showed, might, in the case of wet gun-cotton, rise as high as four miles a second. The construction of fuses—electrical and other—also engaged his attention, and his knowledge of blasting powders and methods of blasting was of great advantage to the Royal Commission on Accidents in Mines, of which he was appointed one of the scientific members at its constitution in 1879.

When mineral oils came into use as a means of domestic illumination it was soon found necessary to frame regulations for their storage, because some of the constituents of the mixture termed petroleum are so volatile that at ordinary temperatures it may give off inflammable vapours which in certain conditions become explosive. The first Petroleum Act, passed in 1862, defined as dangerous, and placed restrictions on the sale of any petroleum that gave off vapour at a lower temperature than 100° F., but as it omitted to specify any method of testing the oil, it was practically inoperative. During the next few years Sir Frederick Abel and other chemists investigated the matter, and as a result, in 1868, the second Petroleum Act was passed, legalising the Abel "open-test" apparatus. In time it was recognised not only that the indications of this instrument were inherently untrustworthy, but that it lent itself to manipulation on the part of an operator who had some motive for not discovering the truth. In 1875, therefore, the whole question was referred to Sir Frederick Abel. He reported that, though the established flashing-point of 100° was calculated to afford adequate protection to the public, the method by which it was ascertained was not satisfactory, and at the same time, as an improvement, he brought forward the Abel "close-test" instrument. This was legalised in 1879, and has been the British standard ever since, besides being adopted, with or without modification, by several other countries. The flash-point of a given oil as determined by the new apparatus not being the same as that shown by the old one, it became necessary to fix the relations between the two. To this end a thousand samples of oil were tested by both methods, and it was found that on the average the temperature at which the oil vapour flashed was 27° lower with the close than with the open test. Hence, the flash-point of 73° was adopted as the equivalent of the 100° prescribed under the old system, and has been adhered to ever since. Agitations (it is to be feared not always inspired by pure regard for the public safety) have from time to time been started to effect an alteration, but without success. The first condition for reducing the annual tale of accidents would rather appear to be due attention to common-sense principles of lamp construction and management, such as are embodied in Sir Frederick Abel's reports to the Metropolitan Board of Works and the Chief Inspector of Explosives.

In 1885 Sir Frederick Abel rendered valuable service as a member of the Council of the Inventions Exhibition, and on the formation of the Imperial Institute, two years later, he was appointed organising secretary and general director. The duties of this office he continued to perform, in later years receiving no pecuniary reward for his services, until the end of his life, and practically until the end of the existence of the Imperial Institute as an independent entity; for it was only in July last that the Royal Assent was given to an Act, which will probably become operative at the beginning of next year, for the transference of the Institute to the control of the Board of Trade. The tenure of this arduous and difficult position did not prevent him from undertaking a great deal of other scientific and official work. He ceased to be Chemist to the War Office in 1888, but immense labour was involved in the investigations of the Special Committee on Explosives, which lasted till 1891. As a prominent member of the Goldsmiths' Company he took considerable interest in the subjects of technical education and original research, and was instrumental in establishing

at the Imperial Institute well-equipped research laboratories in connection with its scientific and technical department.

Sir Frederick Abel received recognition of his scientific work from many quarters. Elected a Fellow of the Royal Society in 1860, he was awarded a Royal medal by that body in 1887. At various times he served as President of the British Association, the Chemical Society, the Institute of Chemistry, the Society of Chemical Industry, and the Institution of Electrical Engineers. Of the Iron and Steel Institute he was elected President in 1891, as a recognition of his scientific contributions to the chemistry of iron and steel, and he was awarded its Bessemer medal in 1897. The University of Cambridge, in which he filled the office of Rede Lecturer, conferred the honorary degree of D.Sc. on him in 1888, and Oxford that of D.C.L. in 1883. He was made C.B. in 1877, a knight in 1883, K.C.B. in 1891, a baronet in 1893, and G.C.V.O. in 1901.

He was twice married, first to Sarah, daughter of Mr. James Blanch, of Bristol, and secondly to Giulietta de la Feuillade, but he has left no heir to the baronetcy.—*The Times*, September 8, 1902.

NOTICES OF BOOKS.

Chemicals and Allied Products. Census Bulletin. By C. E. MUNROE and T. M. CHATARD. Washington. 1902.

THIS volume of statistics is issued in connection with the twelfth census of the United States, and provides a complete account of the present state of chemical industries in the country. Some differences have been made from the reports of 1890 to eliminate certain defects, but care has been taken to preserve the basis of comparison with previous censuses. A short description of methods and processes is attached to the tables of statistics referring to each group of products, and in cases of innovations of importance these descriptive portions are fairly complete; for instance, the contact method for the preparation of sulphuric acid receives special attention, and Knietsch's paper on the subject, which recently appeared in the *Berichte der Deutschen Gesellschaft*, is abstracted. Considerable space is devoted to electro-chemistry, a branch of the science which has not been included in any previous census, as it has practically sprung into existence within the last ten years. A useful digest of United States patents relating to chemical industries (products and processes) is added.

Die Gewinnung des Aluminiums und dessen Bedeutung für Handel und Industrie. ("The Preparation of Aluminium and its Commercial and Industrial Importance"). By ADOLPHE MINET. Halle-a-S.: William Knapp. 1902.

THIS volume is the second of a series of monographs on applied electro-chemistry; in it are described the most recent developments in processes for the preparation of aluminium, and it is well illustrated. Purely chemical methods are not neglected, and no modifications of detail are allowed to pass unnoticed. A full account of Minet's process, which is employed on a large scale, is given, and is specially interesting in view of the fact that there is some difficulty in obtaining trustworthy descriptions of technical processes. The uses to which aluminium may be put are also discussed in the volume, and the characters and properties of the most important alloys. The text has been rendered into German by Dr. Emil Abel, of Vienna. This work must be accorded praise as a careful compilation; it will, no doubt, be found of considerable value by experts.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 4, July 28, 1902.

Reduction of Nitrogen Derivatives by the Method of Direct Hydrogenation in Contact with Finely-divided Metals.—Paul Sabatier and J. B. Senderens.—In a former paper the authors described a general method of direct hydrogenation which can be used in many cases to transform organic nitrogen derivatives into the corresponding amine derivatives. Nitrobenzene, nitrotoluene, and nitronaphthalenes are thus regularly changed into aniline, toluidine, and naphthylamine. They now extend the application of this reaction to the analogous case of nitronaphthalene and the formenic nitrogen derivatives, nitromethane and nitroethane.

Precipitation of Chlorides and Bromides of Cadmium, Mercury, and Tin by means of Sulphuric Acid.—Georges Viard.—An excess of concentrated sulphuric acid precipitates from their solutions the chlorides and bromides of cadmium, mercury, and tin. These salts can be recognised by the reaction described by the author in a previous paper (*Comptes Rendus*, July 21, 1902). The mixture of copper sulphate with a large excess of sulphuric acid, which usually gives a yellow precipitate with chlorides and a black one with bromides, only gives white precipitates with chlorides and bromides of cadmium, mercury, and tin.

MISCELLANEOUS.

Schools of Chemistry, &c.—The following additional information of interest to Students has been received:—

THE INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.—The Institute of Chemistry was founded in October, 1877, and incorporated by Royal Charter in June, 1885, (i.) to promote the better education of persons desirous of becoming public and technical analysts and chemical advisers on scientific subjects; (ii.) to examine Candidates, and to grant certificates of competency; and (iii.) to elevate the profession of Consulting and Analytical Chemistry by setting up a high standard of scientific and practical proficiency and by insisting on the observance of strict rules in regard to professional conduct. *The Studentship.*—Every Candidate for admission to the Studentship is required to produce evidence that he is at least seventeen years of age, and that he has passed a Preliminary Examination in subjects of general education, recognised by the Council of the Institute. He must also show that, at the time of making application for registration, he is working at a College or University approved by the Council, or in the laboratory of a Fellow of the Institute, with the object of adopting the profession of Analytical and Consulting Chemistry. *The Intermediate Examination.*—Candidates for admission to the Intermediate Examination are required to produce evidence (i.) of having passed an approved Preliminary Examination in subjects of general education; (ii.) of having regularly attended systematic day courses, in a College or University recognised by the Council, during at least three academic years, in theoretical and practical Chemistry, and courses in Physics, Mathematics, and one of the following subjects, in accordance with the Regulations of the Institute: (i.) Advanced Mathematics; (ii.) Mechanics and Chemical Engineering; (iii.) Metallurgy; (iv.) Geology and Mineralogy; (v.) Physiology; (vi.) Bacteriology; and (iii.) of having satisfactorily passed the Class Examinations in all the subjects required to be taken. As an alternative in the matter of training (ii.), a candidate may take two years' training in a recognised Institution as indicated above, and work systematically for two other years in the laboratory of a Fellow of the Institute. A

Candidate who has taken a Degree in Science (including Inorganic and Organic Chemistry, and Physics in the Degree Examination, Mathematics having been also passed in at either the Final or Intermediate University Examination) in a University recognised by the Council, is eligible for admission to the Intermediate Examination of the Institute. Holders of certain Degrees or Diplomas are exempt from passing the Intermediate, and, by virtue of their qualifications, are eligible for admission to the Final Examination direct. *The Final Examination for the Associateship (A.I.C.).*—Any Candidate who has passed the Intermediate Examination of the Institute, or who is entitled to claim exemption from passing the Intermediate Examination, is eligible for admission to the Final Examination. *Fellowship (F.I.C.).*—For admission to the Fellowship, an Associate is required to have been registered for three years, and to have been continuously engaged during that period in the study and practical work of Applied Chemistry in a manner satisfactory to the Council. Full particulars are given in "The Book of Regulations for the Admission of Students, Associates, and Fellows," which may be obtained on application to Messrs. Blundell, Taylor, and Co., 173, Upper Thames Street, London, E.C. Price One Shilling.

UNIVERSITY OF BIRMINGHAM.—*Metallurgy*: Professor, Thomas Turner, M.Sc., A.R.S.M., F.I.C. Three courses of Lectures are given during the year—

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- (b) Thirty lectures for Senior Metallurgical Students: Fee, £1 12s. 6d.
- (c) A Special Course of Metallurgical Lectures and Laboratory Work for Engineering Students. Fee, £4 4s.

A course of Lectures and Laboratory Work on Determinative Mineralogy will also be given. Fee, £3 3s. Pending the completion of the new Metallurgical Laboratories and Workshops now in course of erection on the Burnbrook site, practical instruction will be conducted in the laboratories of the Municipal Technical School, which have been specially designed and fully equipped for all forms of metallurgical instruction.

BATTERSEA POLYTECHNIC.—Principal, Mr. S. H. Wells, A.M.I.C.E., A.M.I.M.E. Chemical Department: Head of Department, Mr. John Wilson, M.Sc.; Assistant Lecturers and Demonstrators, J. L. White, M.Sc., H. G. Wayling, B.Sc., J. Hart-Smith, A.R.C.S., A.I.C., and B. C. Folkinghorne, B.Sc., F.C.S. Lecturer in Paper Testing, E. J. Stallybrass. Lecturer in Paper Making, J. Clayton Beadle, F.I.C., F.C.S. Lecturer in Gas Manufacture, J. G. Taplay. Steward and Lecture Assistant, A. E. Dawe. The Chemical Department provides complete courses of instruction in Inorganic and Organic Chemistry, Theoretical and Practical, the courses extending over four years for Evening Students, three years for Day Students, in preparation for the London B.Sc. and various professional examinations. Special courses are also given in Gas Analysis, Gas Manufacture, Paper and Pulp Testing, Paper Making, Assaying, Saccharimetry, Oils, Fats, Soaps, and Candles, Chemistry of Foods, Fuel Analysis, &c. The Senior Day Department provides complete courses of instruction for intending works chemists and general analysts. Students can be admitted for Research and other special work. Recent additions to the equipment comprise an accurate gas analysis machine for research purposes and a small electric furnace.

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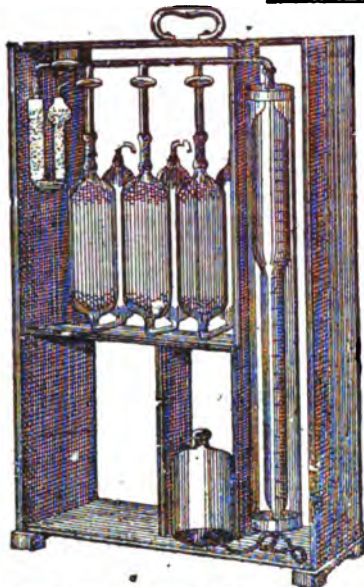
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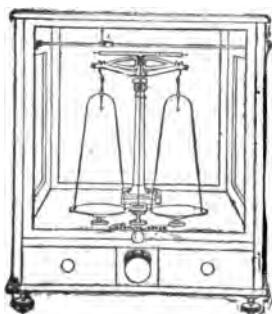
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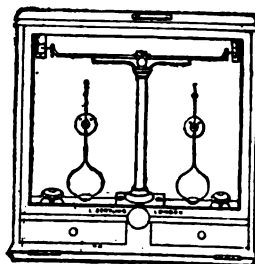


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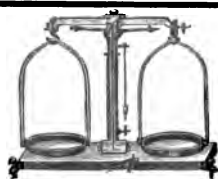
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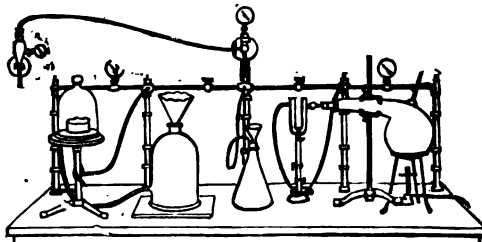
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BRITISH ASSOCIATION FOR THE ADVANCEMENT OF SCIENCE.

BELFAST, 1902.

INAUGURAL ADDRESS OF THE PRESIDENT,
PROF. JAMES DEWAR, M.A., LL.D., D.Sc., F.R.S.

(Continued from p. 132).

History of Cold and the Absolute Zero.

It was Tyndall's good fortune to appear before you at a moment when a fruitful and comprehensive idea was vivifying the whole domain of scientific thought. At the present time no such broad generalisation presents itself for discussion, while, on the other hand, the number of specialised studies has enormously increased. Science is advancing in so broad a front by the efforts of so great an army of workers that it would be idle to attempt within the limits of an address to the most indulgent of audiences anything like a survey of chemistry alone. But I have thought it might be instructive, and perhaps not uninteresting, to trace briefly in broad outline the development of that branch of study with which my own labours have been recently more intimately connected—a study which I trust I am not too partial in thinking is as full of philosophical interest as of experimental difficulty. The nature of heat and cold must have engaged thinking men from the very earliest dawn of speculation upon the external world; but it will suffice for the present purpose if, disregarding ancient philosophers and even mediæval alchemists, we take up the subject where it stood after the great revival of learning, and as it was regarded by the father of the inductive method. That this was an especially attractive subject to Bacon is evident from the frequency with which he recurs to it in his different works, always with lamentation over the inadequacy of the means at disposal for obtaining a considerable degree of cold. Thus in the chapter in the *Natural History*, "*Sylva Sylvarum*," entitled "Experiments in consort touching the Production of Cold," he says, "The production of cold is a thing very worthy of the inquisition both for the use and the disclosure of causes. For heat and cold are nature's two hands whereby she chiefly worketh, and heat we have in readiness in respect of the fire, but for cold we must stay till it cometh or seek it in deep caves or high mountains, and when all is done we cannot obtain it in any great degree, for furnaces of fire are far hotter than a summer sun, but vaults and hills are not much colder than a winter's frost." The great Robert Boyle was the first experimentalist who followed up Bacon's suggestions. In 1682 Boyle read a paper to the Royal Society on "New Experiments and Observations touching Cold, or an Experimental History of Cold," published two years later in a separate work. This is really a most complete history of everything known about cold up to that date, but its great merit is the inclusion of numerous experiments made by Boyle himself on frigorific mixtures, and the general effects of such upon matter. The agency chiefly used by Boyle in the conduct of his experiments was the glaciating mixture of snow or ice and salt. In the course of his experiments he made many important observations. Thus he observed that the salts which did not help the snow or ice to dissolve faster gave no effective freezing. He showed that water in becoming ice expands by about one-ninth of its volume, and bursts gun-barrels. He attempted to counteract the ex-

pansion and prevent freezing by completely filling a strong iron ball with water before cooling; anticipating that it might burst the bottle by the stupendous force of expansion, or that if it did not, then the ice produced might under the circumstances be heavier than water. He speculated in an ingenious way on the change of water into ice. Thus he says, "If cold be but a privation of heat through the recess of that ethereal substance which agitated the little eel-like particles of the water and thereby made them compose a fluid body, it may easily be conceived that they should remain rigid in the postures in which the ethereal substance quitted them, and thereby compose an unfluid body like ice; yet how these little eels should by that recess acquire as strong an endeavour outwards as if they were so many little springs and expand themselves with so stupendous a force, is that which does not so readily appear." The greatest degree of adventitious cold Boyle was able to produce did not make air exposed to its action lose a full tenth of its own volume, so that, in his own words, the cold does not "weaken the spring by anything near so considerable as one would expect." After making this remarkable observation and commenting upon its unexpected nature, it is strange Boyle did not follow it up. He questions the existence of a body of its own nature supremely cold, by participating in which all other bodies obtain that quality, although the doctrine of a *primum frigidum* had been accepted by many sects of philosophers; for, as he says, "if a body being cold signify no more than its not having its sensible parts so much agitated as those of our sensorium, it suffices that the sun or the fire or some other agent, whatever it were, that agitated more vehemently its parts before, does either now cease to agitate them, or agitates them but very remissly, so that till it be determined whether cold be a positive quality or but a privative it will be needless to contend what particular body ought to be esteemed the *primum frigidum*." The whole elaborate investigation cost Boyle immense labour, and he confesses that he "never handled any part of natural philosophy that was so troublesome and full of hardships." He looked upon his results but as a "beginning" in this field of inquiry, and for all the trouble and patience expended he consoled himself with the thought of "men being oftentimes obliged to suffer as much wet and cold and dive as deep to fetch up sponges as to fetch up pearls." After the masterly essay of Boyle, the attention of investigators was chiefly directed to improving thermometrical instruments. The old air thermometer of Galileo being inconvenient to use, the introduction of fluid thermometers greatly aided the inquiry into the action of heat and cold. For a time great difficulty was encountered in selecting proper fixed points on the scales of such instruments, and this stimulated men like Huygens, Newton, Hooke, and Amontons to suggest remedies and to conduct experiments. By the beginning of the eighteenth century the freezing-point and the boiling-point of water were agreed upon as fixed points, and the only apparent difficulties to be overcome were the selection of the fluid, accurate calibration of the capillary tube of the thermometer, and a general understanding as to scale divisions. It must be confessed that great confusion and inaccuracy in temperature observations arose from the variety and crudeness of the instruments. This led Amontons in 1702-3 to contribute two papers to the French Academy which reveal great originality in the handling of the subject, and which, strange to say, are not generally known. The first discourse deals with some new properties of the air and the means of accurately ascertaining the temperature in any climate. He regarded heat as due to a movement of the particles of bodies, though he did not in any way specify the nature of the motion involved; and as the general cause of all terrestrial motion, so that in its absence the earth would be without movement in its smallest parts. The new facts he records are observations on the spring or pressure of air brought about by the action of heat. He

shows that different masses of air measured at the same initial spring or pressure, when heated to the boiling-point of water, acquire equal increments of spring or pressure, provided the volume of the gas be kept at its initial value. Further, he proves that if the pressure of the gas before heating be doubled or tripled, then the additional spring or pressure resulting from heating to the boiling-point of water is equally doubled or tripled. In other words, the ratio of the total spring of air at two definite and steady temperatures and at constant volume is a constant, independent of the mass or the initial pressure of the air in the thermometer. These results led to the increased perfection of the air thermometer as a standard instrument, Amontons' idea being to express the temperature at any locality in fractions of the degree of heat of boiling water. The great novelty of the instrument is that temperature is defined by the measurement of the length of a column of mercury. In passing, he remarks that we do not know the extreme of heat and cold, but that he has given the results of experiments which establish correspondences for those who wish to consider the subject. In the following year Amontons contributed to the Academy a further paper extending the scope of the inquiry. He there pointed out more explicitly that as the degrees of heat in his thermometer are registered by the height of a column of mercury, which the heat is able to sustain by the spring of the air, it follows that the extreme cold of the thermometer will be that which reduces the air to have no power of spring. This, he says, will be a much greater cold than what we call "very cold," because experiments have shown that if the spring of the air at boiling-point is 73 inches, the degree of heat which remains in the air when brought to the freezing-point of water is still very great, for it can still maintain the spring of 51½ inches. The greatest climatic cold on the scale of units adopted by Amontons is marked 50, and the greatest summer heat 58, the value for boiling water being 73, and the zero being 52 units below the freezing-point. Thus Amontons was the first to recognise that the use of air as a thermometric substance led to the inference of the existence of a zero of temperature, and his scale is nothing else than the absolute one we are now so familiar with. It results from Amontons' experiments that the air would have no spring left if it were cooled below the freezing-point of water to about 2½ times the temperature range which separates the boiling-point and the freezing-point. In other words, if we adopt the usual centennial difference between these two points of temperature as 100 degrees, then the zero of Amontons' air thermometer is *minus* 240 degrees. This is a remarkable approximation to our modern value for the same point of *minus* 273 degrees. It has to be confessed that Amontons' valuable contributions to knowledge met with that fate which has so often for a time overtaken the work of too-advanced discoverers; in other words, it was simply ignored, or in any case not appreciated by the scientific world either of that time or half a century later. It is not till Lambert, in his work on "Pyrométrie" published in 1779, repeated Amontons' experiments and endorsed his results that we find any further reference to the absolute scale or the zero of temperature. Lambert's observations were made with the greatest care and refinement, and resulted in correcting the value of the zero of the air scale to *minus* 270 degrees as compared with Amontons' *minus* 240 degrees. Lambert points out that the degree of temperature which is equal to zero is what one may call absolute cold, and that at this temperature the volume of the air would be practically nothing. In other words, the particles of the air would fall together and touch each other, and become dense like water; and from this it may be inferred that the gaseous condition is caused by heat. Lambert says that Amontons' discoveries had found few adherents because they were too beautiful and advanced for the time in which he lived.

About this time a remarkable observation was made by Professor Braun at Moscow, who, during the severe winter

of 1759, succeeded in freezing mercury by the use of a mixture of snow and nitric acid. When we remember that mercury was regarded as quite a peculiar substance possessed of the essential quality of fluidity, we can easily understand the universal interest created by the experiment of Braun. This was accentuated by the observations he made on the temperature given by the mercury thermometer, which appeared to record a temperature as low as *minus* 200° C. The experiments were soon repeated by Hutchins at Hudson's Bay, who conducted his work with the aid of suggestions given him by Cavendish and Black. The result of the new observations was to show that the freezing-point of mercury is only *minus* 40° C., the errors in former experiments having been due to the great contraction of the mercury in the thermometer in passing into the solid state. From this it followed that the enormous natural and artificial colds which had generally been believed in had no proved existence. Still the possible existence of a zero of temperature very different from that deduced from gas thermometry had the support of such distinguished names as those of Laplace and Lavoisier. In their great memoir on "Heat," after making what they consider reasonable hypotheses as to the relation between specific heat and total heat, they calculate values for the zero which range from 1500° to 3000° below melting ice. On the whole, they regard the absolute zero as being in any case 600° below the freezing-point. Lavoisier, in his "Elements of Chemistry" published in 1792, goes further in the direction of indefinitely lowering the zero of temperature when he says, "We are still very far from being able to produce the degree of absolute cold, or total deprivation of heat, being unacquainted with any degree of coldness which we cannot suppose capable of still further augmentation: hence it follows we are incapable of causing the ultimate particles of bodies to approach each other as near as possible, and thus these particles do not touch each other in any state hitherto known." Even as late as the beginning of the nineteenth century we find Dalton, in his new system of "Chemical Philosophy," giving ten calculations of this value, and adopting finally as the natural zero of temperature *minus* 3000° C.

In Black's lectures we find that he takes a very cautious view with regard to the zero of temperature, but as usual is admirably clear with regard to its exposition. Thus he says, "We are ignorant of the lowest possible degree or beginning of heat. Some ingenious attempts have been made to estimate what it may be, but they have not proved satisfactory. Our knowledge of the degrees of heat may be compared to what we should have of a chain, the two ends of which were hidden from us, and the middle only exposed to our view. We might put distinct marks on some of the links, and number the rest according as they are nearest to or further removed from the principal links; but not knowing the distance of any links from the end of the chain we could not compare them together with respect to their distance, or say that one link was twice as far from the end of the chain as another." It is interesting to observe, however, that Black was evidently well acquainted with the work of Amontons, and strongly supports his inference as to the nature of air. Thus, in discussing the general cause of vaporisation, Black says that some philosophers have adopted the view "that every palpable elastic fluid in nature is produced and preserved in this form by the action of heat. Mr. Amontons, an ingenious member of the late Royal Academy of Sciences, at Paris, was the first who proposed this idea with respect to the atmosphere. He supposed that it might be deprived of the whole of its elasticity and condensed, and even frozen into a solid matter were it in our power to apply to it a sufficient cold; that it is a substance that differs from others by being incomparably more volatile, and which is therefore converted into vapour and preserved in that form by a weaker heat than any that ever happened or can obtain in this globe, and which therefore cannot appear under any other form than

the one it now wears, so long as the constitution of the world remains the same as at present." The views that Black attributes to Amontons have been generally associated with the name of Lavoisier, who practically admitted similar possibilities as to the nature of air; but it is not likely that in such matters Black would commit any mistake as to the real author of a particular idea, especially in his own department of knowledge. Black's own special contribution to low-temperature studies was his explanation of the interaction of mixtures of ice with salts and acids by applying the doctrine of the latent heat of fluidity of ice to account for the frigorific effect. In a similar way Black explained the origin of the cold produced in Cullen's remarkable experiment of the evaporation of ether under the receiver of an air-pump by pointing out that the latent heat of vaporisation in this case necessitated such a result. Thus, by applying his own discoveries of latent heat, Black gave an intelligent explanation of the cause of all the low-temperature phenomena known in his day.

After the gaseous laws had been definitely formulated by Gay-Lussac and Dalton, the question of the absolute zero of temperature, as deduced from the properties of gases, was revived by Clement and Desormes. These distinguished investigators presented a paper on the subject to the French Academy in 1812, which, it appears, was rejected by that body. The authors subsequently elected to publish it in 1819. Relying on what we know now to have been a faulty hypothesis, they deduced from observations on the heating of air rushing into a vacuum the temperature of *minus* 267 degrees as that of the absolute zero. They further endeavoured to show, by extending to lower temperatures the volume or the pressure coefficients of gases given by Gay-Lussac, that at the same temperature of *minus* 267 degrees the gases would contract so as to possess no appreciable volume, or, alternatively, if the pressure was under consideration, it would become so small as to be non-existent. Although full reference is given to previous work bearing on the same subject, yet, curiously enough, no mention is made of the name of Amontons. It certainly gave remarkable support to Amontons' notion of the zero to find that simple gases like hydrogen and compound gases like ammonia, hydrochloric, carbonic, and sulphurous acids should all point to substantially the same value for this temperature. But the most curious fact about this research of Clement and Desormes is that Gay-Lussac was a bitter opponent of the validity of the inferences they drew either from his work or their own. The mode in which Gay-Lussac regarded the subject may be succinctly put as follows:—A quick compression of air to one-fifth volume raises its temperature to 300 degrees, and if this could be made much greater and instantaneous the temperature might rise to 1000 or 2000 degrees. Conversely, if air under five atmospheres were suddenly dilated, it would absorb as much heat as it had evolved during compression, and its temperature would be lowered by 300 degrees. Therefore, if air were taken and compressed to fifty atmospheres or more, the cold produced by its sudden expansion would have no limit. In order to meet this position Clement and Desormes adopted the following reasoning:—They pointed out that it had not been proved that Gay-Lussac was correct in his hypothesis, but that in any case it tacitly involves the assumption that a limited quantity of matter possesses an unlimited supply of heat. If this were the case, then heat would be unlike any other measurable thing or quality. It is, therefore, more consistent with the course of nature to suppose that the amount of heat in a body is like the quantity of elastic fluid filling a vessel, which, while definite in original amount, one may make less and less by getting nearer to a complete exhaustion. Further, to realise the absolute zero in the one case is just as impossible as to realise the absolute vacuum in the other; and as we do not doubt a zero of pressure, although it is unattainable, for the same reason we ought to accept the

reality of the absolute zero. We know now that Gay-Lussac was wrong in supposing the increment of temperature arising from a given gaseous compression would produce a corresponding decrement from an identical expansion. After this time the zero of temperature was generally recognised as a fixed ideal point, but in order to show that it was hypothetical a distinction was drawn between the use of the expressions, zero of absolute temperature and the absolute zero.

The whole question took an entirely new form when Lord Kelvin, in 1848, after the mechanical equivalent of heat had been determined by Joule, drew attention to the great principles underlying Carnot's work on the "Motive Power of Heat," and applied them to an absolute method of temperature measurement, which is completely independent of the properties of any particular substance. The principle was that for a difference of one degree on this scale, between the temperatures of the source and refrigerator, a perfect engine should give the same amount of work in every part of the scale. Taking the same fixed points as for the centigrade scale, and making 100 of the new degrees cover that range, it was found that the degrees not only within that range, but as far beyond as experimental data supplied the means of comparison, differed by only minute quantities from those of Regnault's air thermometer. The zero of the new scale had to be determined by the consideration that when the refrigerator was at the zero of temperature the perfect engine should give an amount of work equal to the full mechanical equivalent of the heat taken up. This led to a zero of 273 degrees below the temperature of freezing water, substantially the same as that deduced from a study of the gaseous state. It was a great advance to demonstrate by the application of the laws of thermodynamics not only that the zero of temperature is a reality, but that it must be located at 273 degrees below the freezing-point of water. As no one has attempted to impugn the solid foundation of theory and experiment on which Lord Kelvin based his thermodynamic scale, the existence of a definite zero of temperature must be acknowledged as a fundamental scientific fact.

Liquefaction of Gases and Continuity of State.

In these speculations, however, chemists were dealing theoretically with temperatures to which they could not make any but the most distant experimental approach. Cullen, the teacher of Black, had indeed shown how to lower temperature by the evaporation of volatile bodies, such as ether, by the aid of the air-pump, and the later experiments of Leslie and Wollaston extended the same principle. Davy and Faraday made the most of the means at command in liquefying the more condensable gases, while at the same time Davy pointed out that they in turn might be utilised to procure greater cold by their rapid re-conversion into the aeriform state. Still the chemist was sorely hampered by the want of some powerful and accessible agent for the production of temperatures much lower than had ever been attained. That want was supplied by Thilorier, who in 1835 produced liquid carbonic acid in large quantities, and further made the fortunate discovery that the liquid could be frozen into a snow by its own evaporation. Faraday was prompt to take advantage of this new and potent agent. Under exhaustion he lowered its boiling-point from *minus* 78° C. to *minus* 110° C., and by combining this low temperature with pressure all the gases were liquefied by the year 1844, with the exception of the three elementary gases—hydrogen, nitrogen, and oxygen, and three compound gases—carbonic oxide, marsh gas, and nitric oxide; Andrews some twenty-five years after the work of Faraday attempted to induce change of state in the uncondensed gases by using much higher pressures than Faraday employed. Combining the temperature of a solid carbonic acid bath with pressures of 300 atmospheres, Andrews found that none of these gases exhibited any appearance of liquefaction in such high states of condensation; but so

far as change of volume by high compression went, Andrews confirmed the earlier work of Natterer by showing that the gases become proportionately less compressible with growing pressure. While such investigations were proceeding, Regnault and Magnus had completed their refined investigations on the laws of Boyle and Gay-Lussac. A very important series of experiments was made by Joule and Kelvin "On the Thermal Effects of Fluids in Motion" about 1862, in which the thermometrical effects of passing gases under compression through porous plugs furnished important data for the study of the mutual action of the gas molecules. No one, however, had attempted to make a complete study of a liquefiable gas throughout wide ranges of temperature. This was accomplished by Andrews in 1869, and his Bakerian Lecture "On the Continuity of the Gaseous and Liquid States of Matter," will always be regarded as an epoch-making investigation. During the course of this research Andrews observed that liquid carbonic acid raised to a temperature of 31°C . lost the sharp concave surface of demarcation between the liquid and the gas, the space being now occupied by a homogeneous fluid which exhibited, when the pressure was suddenly diminished or the temperature slightly lowered, a peculiar appearance of moving or flickering striae, due to great local alterations of density. At temperatures above 31°C . the separation into two distinct kinds of matter could not be effected even when the pressure reached 400 atmospheres. This limiting temperature of the change of state from gas to liquid Andrews called the critical temperature. He showed that this temperature is constant, and differs with each substance, and that it is always associated with a definite pressure peculiar to each body. Thus the two constants, critical temperature and pressure, which have been of the greatest importance in subsequent investigations, came to be defined, and a complete experimental proof was given that "the gaseous and liquid states are only distinct stages of the same condition of matter, and are capable of passing into one another by a process of continuous change."

¶ In 1873 an essay "On the Continuity of the Gaseous and Liquid State," full of new and suggestive ideas, was published by van der Waals, who, recognising the value of Clausius's new conception of the virial in dynamics, for a long-continued series of motions, either oscillatory or changing exceedingly slowly with time, applied it to the consideration of the molecular movements of the particles of the gaseous substance, and after much refined investigation, and the fullest experimental calculation available at the time, devised his well-known equation of continuity. Its paramount merit is that it is based entirely on a mechanical foundation, and is in no sense empiric; we may therefore look upon it as having a secure foundation in fact, but as being capable of extension and improvement. James Thomson, realising that the straight-line breach of continuous curvature in the Andrews isothermals was untenable to the physical mind, propounded his emendation of the Andrews curves—namely, that they were continuous and of S form. We also owe to James Thomson the conception and execution of a three-dimensional model of Andrews' results, which has been of the greatest service in exhibiting the three variables by means of a specific surface afterwards greatly extended and developed by Professor Willard Gibbs. The suggestive work of James Thomson undoubtedly was a valuable aid to van der Waals, for as soon as he reached the point where his equation had to show the continuity of the two states this was the first difficulty he had to encounter, and he succeeded in giving the explanation. He also gave a satisfactory reason for the existence of a minimum value of the product of volume and pressure in the Regnault isothermals. His isothermals, with James Thomson's completion of them, were now shown to be the results of the laws of dynamics. Andrews applied the new equation to the consideration of the coefficients of expansion with temperature and of pressure with tem-

perature, showing that although they were nearly equal, nevertheless they were almost independent quantities. His investigation of the capillarity constant was masterly, and he added further to our knowledge of the magnitudes of the molecules of gases and of their mean free paths. Following up the experiments of Joule and Kelvin, he showed how their cooling coefficients could be deduced, and proved that they vanished at a temperature in each case which is a constant multiple of the specific critical temperature. The equation of continuity developed by van der Waals involved the use of three constants instead of one, as in the old law of Boyle and Charles, the latter being only utilised to express the relation of temperature, pressure, and volume, when the gas is far removed from its point of liquefaction. Of the two new constants one represents the molecular pressure arising from the attraction between the molecules, the other four times the volume of the molecules. Given these constants of a gas, van der Waals showed that his equation not only fitted into the general characters of the isothermals, but also gave the values of the critical temperature, the critical pressure, and the critical volume. In the case of carbonic acid the theoretical results were found to be in remarkable agreement with the experimental values of Andrews. This gave chemists the means of ascertaining the critical constants, provided sufficiently accurate data derived from the study of a few properly distributed isothermals of the gaseous substance were available. Such important data came into the possession of chemists when Amagat published his valuable paper on "The Isothermals of Hydrogen, Nitrogen, Oxygen, Ethylene, &c.," in the year 1880. It now became possible to calculate the critical data with comparative accuracy for the so-called permanent gases oxygen and nitrogen, and this was done by Sarrau in 1882. In the meantime a great impulse had been given to a further attack upon the so-called permanent gases by the suggestive experiments made by Pictet and Cailletet. The static liquefaction of oxygen was effected by Wroblewski in 1883, and thereby the theoretical conclusions derived from van der Waals' equation were substantially confirmed. The liquefaction of oxygen and air was achieved through the use of liquid ethylene as a cooling agent, which enabled a temperature of minus 140 degrees to be maintained by its steady evaporation *in vacuo*. From this time liquid oxygen and air came to be regarded as the potential cooling agents for future research, commanding as they did a temperature of 200 degrees below melting ice. The theoretical side of the question received at the hands of van der Waals a second contribution, which was even more important than his original essay, and that was his novel and ingenious development of what he calls "The Theory of Corresponding States." He defined the corresponding states of two substances as those in which the ratios of the temperature, pressure, and volume to the critical temperature, pressure, and volume respectively were the same for the two substances, and in corresponding states he showed that the three pairs of ratios all coincided. From this a series of remarkable propositions were developed, some new, some proving previous laws that were hitherto only empiric, and some completing and correcting faulty though approximate laws. As examples, he succeeded in calculating the boiling-point of carbonic acid from observations on ether vapour, proved Kopp's law of molecular volumes, and showed that at corresponding temperatures the molecular latent heats of vaporisation are proportional to the absolute critical temperature, and that under the same conditions the coefficients of liquid expansion are inversely proportional to the absolute critical temperature, and that the coefficients of liquid compressibility are inversely proportional to the critical pressure. All these propositions and deductions are in the main correct, though further experimental investigation has shown minor discrepancies requiring explanation. Various proposals have been made to supplement van der Waals' equation so as to bring it into line with experiments, some

being entirely empiric, others theoretical. Clausius, Sarrau, Wroblewski, Battelli, and others attacked the question empirically, and in the main preserved the co-volume (depending on the total volume of the molecules) unaltered while trying to modify the constant of molecular attraction. Their success depended entirely on the fact that, instead of limiting the number of constants to three, some of them have increased them to as many as ten. On the other hand, a series of very remarkable theoretical investigations has been made by van der Waals himself, by Kammerlingh Onnes, Korteweg, Jaeger, Boltzmann, Dieterici, and Riensmann, and others, all directed in the main towards an admitted variation in the value of the co-volume while preserving the molecular attraction constant. The theoretical deductions of Tait lead to the conclusion that a substance below its critical point ought to have two different equations of the van der Waals type, one referring to the liquid, and the other to the gaseous phase. One important fact was soon elicited—namely, that the law of correspondence demanded only that the equation should contain not more than three constants for each body. The simplest extension is that made by Riensmann, in which he increased the pressure for a given mean kinetic energy of the particles inversely in the ratio of the diminution of free volume, due to the molecules possessing linear extension. Berthelot has shown how a "reduced" isothermal may be got by taking two other prominent points as units of measurement instead of the critical coordinates. The most suggestive advance in the improvement of the van der Waals equation has been made by a lady, M^{me}. Christine Meyer. The idea at the base of this new development may be understood from the following general statement:—van der Waals brings the van der Waals surfaces for all substances into coincidence at the point where volume, pressure, and temperature are nothing, and then stretches or compresses all the surfaces parallel to the three axes of volume, pressure, and temperature, until their critical points coincide. But on this plan the surfaces do not quite coincide, because the points where the three variables are respectively nothing are not corresponding points. M^{me}. Meyer's plan is to bring all the critical points first into coincidence, and then to compress or extend all the representative surfaces parallel to the three axes of volume, pressure, and temperature, until the surfaces coincide. In this way, taking twenty-nine different substances, she completely verifies from experiment van der Waals' law of correspondence. The theory of van der Waals has been one of the greatest importance in directing experimental investigation, and in attacking the difficult problems of the liquefaction of the most permanent gases. One of its greatest triumphs has been the proof that the critical constants and the boiling-point of hydrogen theoretically deduced by Wroblewski from a study of the isothermals of the gas taken far above the temperature of liquefaction are remarkably near the experimental values. We may safely infer, therefore, that if hereafter a gas be discovered in small quantity even four times more volatile than liquid hydrogen, yet by a study of its isothermals at low temperature we shall succeed in finding its most important liquid constants, although the isolation of the real liquid may for the time be impossible. It is perhaps not too much to say that as a prolific source of knowledge in the department dealing with the continuity of state in matter, it would be necessary to go back to Carnot's cycle to find a proposition of greater importance than the theory of van der Waals and his development of the law of corresponding states.

It will be apparent from what has just been said that, thanks to the labours of Andrews, van der Waals, and others, theory had again far outrun experiment. We could calculate the constants and predict some of the simple physical characteristics of liquid oxygen, hydrogen, or nitrogen with a high degree of confidence long before any one of the three had been obtained in the static liquid condition permitting of the experimental verification of the theory. This was the more tantalising, because, with

whatever confidence the chemist may anticipate the substantial corroboration of his theory, he also anticipates with almost equal conviction that as he approaches more and more nearly to the zero of absolute temperature, he will encounter phenomena compelling modification, revision, and refinement of formulæ which fairly covered the facts previously known. Just as nearly seventy years ago chemists were waiting for some means of getting a temperature of 100 degrees below melting ice, so ten years ago they were casting about for the means of going 100 degrees lower still. The difficulty, it need hardly be said, increases in a geometrical rather than in an arithmetical ratio. Its magnitude may be estimated from the fact that to produce liquid air in the atmosphere of an ordinary laboratory is a feat analogous to the production of liquid water starting from steam at a white heat, and working with all the implements and surroundings at the same high temperature. The problem was not so much how to produce intense cold as how to save it when produced from being immediately levelled up by the relatively superheated surroundings. Ordinary non-conducting packings were inadmissible because they are both cumbrous and opaque, while in working near the limits of our resources it is essential that the product should be visible and readily handled. It was while puzzling over this mechanical and manipulative difficulty in 1892 that it occurred to me that the principle of an arrangement used nearly twenty years before in some calorimetric experiments, which was based upon the work of Dulong and Petit on radiation, might be employed with advantage as well to protect cold substances from heat as hot ones from rapid cooling. I therefore tried the effect of keeping liquefied gases in vessels having a double wall, the annular space between being very highly exhausted. Experiments showed that liquid air evaporated at only one-fifth of the rate prevailing when it was placed in a similar unexhausted vessel, owing to the convective transference of heat by the gas particles being enormously reduced by the high vacuum. But, in addition, these vessels lend themselves to an arrangement by which radiant heat can also be cut off. It was found that when the inner walls were coated with a bright deposit of silver the influx of heat was diminished to one-sixth the amount entering without the metallic coating. The total effect of the high vacuum and the silvering is to reduce the ingoing heat to about 3 per cent. The efficiency of such vessels depends upon getting as high a vacuum as possible, and cold is one of the best means of effecting the desired exhaustion. All that is necessary is to fill completely the space that has to be exhausted with an easily condensable vapour, and then to freeze it out in a receptacle attached to the primary vessel that can be sealed off. The advantage of this method is that no air-pump is required, and that theoretically there is no limit to the degree of exhaustion that can be obtained. The action is rapid, provided liquid air is the cooling agent, and vapours like mercury, water, or benzol are employed. It is obvious that when we have to deal with such an exceptionally volatile liquid as hydrogen, the vapour filling may be omitted because air itself is now an easily condensable vapour. In other words, liquid hydrogen, collected in such vessels with the annular space full of air, immediately solidifies the air, and thereby surrounds itself with a high vacuum. In the same way, when it shall be possible to collect a liquid boiling on the absolute scale at about 5 degrees, as compared with the 20 degrees of hydrogen, then you might have the annular space filled with the latter gas to begin with, and yet get directly a very high vacuum, owing to the solidification of the hydrogen. Many combinations of vacuum vessels can be arranged, and the lower the temperature at which we have to operate the more useful they become. Vessels of this kind are now in general use, and in them liquid air has crossed the American continent. Of the various forms, that variety is of special importance which has a spiral tube joining the bottom part of the walls, so that any

liquid gas may be drawn off from the interior of such a vessel. In the working of regenerative coils such a device becomes all-important, and such special vessels cannot be dispensed with for the liquefaction of hydrogen.

In the early experiments of Pictet and Cailletet, cooling was produced by the sudden expansion of the highly compressed gas preferably at a low temperature, the former using a jet that lasted for some time, the latter an instantaneous adiabatic expansion in a strong glass tube. Neither process was practicable as a model of producing liquid gases, but both gave valuable indications of partial change into the liquid state by the production of a temporary mist. Linde, however, saw that the continuous use of a jet of highly compressed gas, combined with regenerative cooling, must lead to liquefaction on account of what is called the Kelvin-Joule effect; and he succeeded in making a machine, based on this principle, capable of producing liquid air for industrial purposes. These experimenters had proved that, owing to molecular attraction, compressed gases passing through a porous plug or small aperture were lowered in temperature by an amount depending on the difference of pressure and inversely as the square of the absolute temperature. This means that for a steady difference of pressure the cooling is greater the lower the temperature. The only gas that did not show cooling under such conditions was hydrogen. Instead of being cooled it became actually hotter. The reason for this apparent anomaly in the Kelvin-Joule effect is that every gas has a thermometric point of inversion above which it is heated and below which it is cooled. This inversion point, according to van der Waals, is six and three-quarter times the critical point. The efficiency of the Linde process depends on working with highly compressed gas well below the inversion temperature, and in this respect this point may be said to take the place of the critical one, when in the ordinary way direct liquefaction is being effected by the use of specific liquid cooling agents. The success of both processes depends upon working within a certain temperature range, only the Linde method gives us a much wider range of temperature within which liquefaction can be effected. This is not the case if, instead of depending on getting cooling by the internal work done by the attraction of the gas molecules, we force the compressed gas to do external work as in the well-known air machines of Kirk and Coleman. Both these inventors have pointed out that there is no limit of temperature, short of liquefaction of the gas in use in the circuit, that such machines are not capable of giving. While it is theoretically clear that such machines ought to be capable of maintaining the lowest temperatures, and that with the least expenditure of power, it is a very different matter to overcome the practical difficulties of working such machines under the conditions. Coleman kept a machine delivering air at minus 83 degrees for hours, but he did not carry his experiments any further. Recently Monsieur Claude, of Paris, has, however, succeeded in working a machine of this type so efficiently that he has managed to produce one litre of liquid air per horse power expended per hour in the running of the engine. This output is twice as good as that given by the Linde machine, and there is no reason to doubt that the yield will be still further improved. It is clear, therefore, that in the immediate future the production of liquid air and hydrogen will be effected most economically by the use of machines producing cold by the expenditure of mechanical work.

(To be continued).

Appointment.—The Governors of the Northern Polytechnic Institute have appointed Mr. W. H. Mills, M.A., Ph.D., Fellow of Jesus College, Cambridge, as Head of the Chemical Department, to succeed Mr. H. C. L. Bloxam, who resigned the position in order to take up an appointment in Cape Town.

ADDRESS TO THE CHEMICAL SECTION OF THE BRITISH ASSOCIATION. BELFAST, 1902.

By EDWARD DIVERS, M.D., D.Sc., F.R.S., V.P.C.S.,
Emeritus Professor of Chemistry in the Imperial University of Tokyo,
Japan.

President of the Section.

THE ATOMIC THEORY WITHOUT HYPOTHESIS.

IN opening the Chemical Section of the British Association in this city and in the halls of the Queen's College, my first words must be those of reverence for the memory of Thomas Andrews, for so many years the Professor of Chemistry in this College, whose investigations into the properties of gases—above all, those which resulted in the recognition and determination of the critical pressure and temperature of carbonic anhydride—have become a part of the foundation of the Kinetic Theory of Gases. At the meeting of the British Association here in 1852 Andrews was President of this Section, and again at the meeting in Edinburgh in 1871.

Since the meeting last year another distinguished chemist, formerly professor in one of the Queen's Colleges, Maxwell Simpson, has also passed away. He, too, acted as President of this Section, namely, at the meeting in Dublin in 1878. The work by which Simpson's name will ever be recalled is more especially that upon the synthesis of polybasic organic acids.

One other name must not be left unmentioned in this Address: it is that of a long-time Fellow of the Chemical Society who has been intimately connected with the British Association—I mean that of George Griffith, the genial and most effective Assistant General Secretary of the Association for so many years, who died four months ago. He had visited Belfast in the spring and made the preliminary arrangements with the Local Committee for this meeting. He joined the Chemical Society in 1859—just one year before I did—and remained a Fellow until his death.

It is now almost a century ago since John Dalton made known to the world his theory of the nature of chemical combination by the publication of a table of atomic weights. He had been occupying himself for some years with the study of the physical properties and atomic constitution of gases before he was led to extend the notion of the atom to chemical phenomena, and thus to form that conception which was to become celebrated as the atomic theory. In his laboratory note-books, preserved from 1802 onwards, the publication and analysis of which we owe to Sir Henry Roscoe and Dr. Harden, no reference is made to the theory till 1803, but we may well believe with Henry that it was already in Dalton's mind just a hundred years ago. But however that may have been, it seems fitting in a year so closely approaching the centennial of its publication as the present that the occupier of this Chair should address his audience on a subject of such general interest and importance as the atomic theory, if indeed there remains anything to be said on a subject which has so long and so fully engaged attention.

I dare not assert that I have found anything actually novel to bring before you with regard to the atomic theory, but I may say that there has certainly long seemed to me to exist the need to treat it as being a true theory instead of as an hypothesis, and to teach it and discuss it accordingly.

In thus setting forth what appears to me to be the proper form of the atomic theory, I shall have, at the risk of overtaxing your patience, to re-state and examine most of the fundamental and familiar principles of our science in order to illustrate and justify the view I take. Not only this, but in order as directly and briefly as possible to meet the objection that, whatever the atomic theory may be, it cannot be introduced to the student of chemical philosophy in another form than that now in use, I

shall sometimes have to adopt, in order to show what can be done, a didactic method which, in most other circumstances, would be quite inexcusable before so distinguished an assembly.

The atomic theory of chemistry stands unsurpassed for the way in which it has fulfilled the purpose of every great theory, that of giving intellectual mastery of the phenomena of which it treats. But in the form in which it was enunciated, and still is universally expressed and accepted, it has the defect of resting upon a metaphysical basis, namely, upon the ancient hypothesis that bodies are not continuous in texture, but consist of discrete, ultra-minute particles whose properties, if known, would account for those of the bodies themselves. Hence it has happened that, despite the light it throws upon the relations of chemical phenomena and the simple means it affords of expressing these relations, this theory has always been regarded with misgiving, and failed to achieve that explicit recognition which its abounding merit calls for. Indeed, the desire has been expressed to see the time when something on a more solid foundation shall have taken its place.

Now, it is not my intention to discuss the merits or demerits of the atomic hypothesis, which can indeed no longer be treated as a merely metaphysical speculation. What I would do to-day is to impress upon you that, in spite of all that has been said and written about the atomic hypothesis in connection with chemistry, the atomic theory propounded by Dalton and adopted, implicitly at least, by all chemists, is not founded upon the metaphysical conception of material discontinuity, and is not explained or illuminated by it. For if that should be the case there will no longer exist any grounds for hesitation in accepting the theory quite explicitly, and then the anomalous condition of things will be removed of a theory being in universal use without its truth being freely and openly admitted. For the sake of clearness, it is convenient to restrict the term "atomic hypothesis" to the old metaphysical view of the discontinuity of matter whilst applying the term "atomic theory" to the current elaborated form of the Daltonian theory; this distinction is adhered to in the present Address.

In the peroration to his admirable discourse upon atomic weights or masses delivered before the Chemical Society in 1892 as the Stas Memorial Lecture, Professor Mallet, F.R.S., said:—"By the chemist at his balance the arm of reason is directed into those regions of almost inconceivable minuteness, which lie as far beyond the reach of the most powerful microscope as that carries us beyond the reach of the naked eye, quite as impressively as that same arm is stretched forth by the astronomer at his divided circle to reach and to weigh the mighty planets that shine in the remotest regions of our solar system." On two occasions I have heard the same comparison between the chemist and the astronomer made by Lord Kelvin when he was in the company of chemists; and undoubtedly both these high authorities have only then expressed the general view as to the nature of the domain of the chemist. Yet I venture to question whether there is anything in the ways and works of the chemist to support such a view and give point to Mallet and Kelvin's comparison. If, indeed, chemistry is a science which rests upon the atomic hypothesis and, therefore, would cease to exist in the form into which it has developed, should matter prove to be continuous and not discrete, nothing can be said against the view that it is a science of the minute. But I am sure there can be no one ready to maintain that, if the hypothesis of the atomic constitution of substances were an unfounded one, the atomic theory would have been a discovery of no great importance; and Dalton himself, instead of being the founder of the chemistry of to-day, have been little more than the discoverer of the law of multiple proportions. If that cannot be maintained, what, then, becomes of this conception of chemistry as dealing with the minute? So far as comparison can be made between the operations of

the astronomer and the chemist, it is the former and not the latter who, as a matter of fact, deals with the almost infinitely minute. For if, indeed, the chemist often works upon comparatively small amounts of substances, and, consequently, with very sensitive balances, that is, as we all know, only for reasons of economy of time, materials, and apparatus; otherwise he works on the largest possible scale, with the object of attaining to the highest degree of accuracy and perfection. The astronomer, on the other hand, has, perforce, to deal with the smallest visible things in nature, the nearest approach there is to geometrical points, those fixed points of light in the heavens which are only known through scientific investigation to be other than what they seem to be. It is, therefore, only as interpreted by the atomic hypothesis that chemistry can be said to deal with the minute.

When the atomic theory is expounded in the usual way it is commonly and correctly stated that, on the assumption that substances consist of minute indivisible particles having weights or masses bearing the ratios of the combining numbers assigned to them, the laws of chemical combination by weight necessarily follow, and are thereby explained. But then the converse is not true—that, because chemical combination obeys the well-known laws, substances consist of discrete particles. Nor does the assumption of the truth of the atomic hypothesis afford any real explanation of the facts expressed by the laws of chemical combination, or more comprehensively by the atomic theory, when that theory is given in non-hypothetical terms. It is just as difficult to see why the atoms should possess the weights on chemical grounds assigned to them, as to see why substances interact in the proportions that they do; that they do so is, in either case, an ultimate fact, for which no explanation has presented itself. The atomic hypothesis masks this ignorance and deadens inquisitiveness. Notwithstanding all this, which is incontrovertible, it is certainly a common opinion that in chemistry we investigate the minute and intimate constitution of things.

But if, after all, chemistry does not deal with the minute, or, rather, if it has no concern with the magnitude of single bodies or their molecules; if the atomic hypothesis is not the foundation of, or necessary to, the atomic theory, then it is certainly most desirable and important that the theory of chemistry, which, with all its modern developments, I take to be indisputably the atomic theory of Dalton, should be held and expounded without any reference to the physical constitution of matter, in so far as that remains unknown. The opinion that chemical theory should be developed without reference to the atomic hypothesis has indeed all along been held by many eminent chemists; but then the dilemma appears to have presented itself to them, that either the atomic hypothesis must be granted, or the atomic theory must be dispensed with, since it falls with the hypothesis. That dilemma I do not recognise, and the practice of chemists shows beyond doubt that it is always ignored. Investigators use the theory, whether they admit it or not; teachers of the science find it indispensable to their task, however much they may deprecate, and rightly so, unreserved acceptance of the atomic hypothesis as true.

Refusing to commit themselves to belief in the hypothesis, chemists have thought from the first to escape the adoption of the atomic theory by putting Dalton's discovery into something like these words:—Numbers, called proportional or combining numbers, can be assigned to the chemical elements—one to each—which will express all the ratios of the weights or masses in which substances interact and combine together. Perhaps the atomic theory is here successfully set aside by expressing what is an actuality as an unaccounted for possibility. But then those who use any such mode of expressing the facts, without reference to the theory, never fail also to adopt the doctrine of equivalents, and thus, by this double act, implicitly give in their adherence to the theory.

Divested of all reference to the physical constitution of

matter, the atomic theory is that the quantities of substances which interact in single chemical changes are equal to one another,—as truly equal in one way as equal masses are in another,—and, therefore, that chemical interaction is a measure of quantity of unlike substances, distinct from, and independent of, dynamical or mass measurement.

Dalton, indeed, did not express himself in any such terms, his mind being fully possessed with the ancient and current belief upon which he framed his theory that substances are made up of minute, discrete particles. But it is clear enough that his theory was that of the existence of another order of equality between substances than that of weight. Up to this time, the weight or mass of every ultimate particle of any substance whatever appears to have been assumed to be the same, the atoms being alike in every way. That assumption is still made by many thinkers, chemists among them; we meet it, for example, in the different forms of the hypothesis that the elements are all, in some way, physically compounded of a universal and only true element, as in Prout's hypothesis. Dalton saw things differently, and recognised that, on the assumption of substances being constituted of particles which never subdivide, weight or mass cannot be the same for every such particle, except in the case of those of any one simple substance. Therefore, having given some numbers showing what he believed to be the respective weights of the atoms of several simple substances, taking that of hydrogen as of unit-weight, he proceeded at once to invent symbols for these atoms to indicate, not only their distinctness in kind, but above all things their indivisibility and their equality, properties which the use of their atomic numbers would have inadvertently concealed or even apparently denied, and could never have expressed or connoted.

It was only in this immediate invention and use of chemical symbols that Dalton's conception found clear expression; and again it is by the universal adoption of such symbols that chemists have shown their real acceptance of the atomic theory, even while displaying, not infrequently, their scepticism as to its truth. The replacement by Berzelius of Dalton's marked circles for atomic symbols by letters which should recall the names of the substances was in a way a great improvement, but it has had the serious consequence of causing chemical symbols to be usually first brought under notice merely as serviceable abbreviations for the names of the elements, and only then described as representing their atomic quantities. Now, evidently, what the character used as symbol shall be is, theoretically considered, but a petty detail; the vital point is what the character symbolises, and that is the atom. It does not symbolise the name; it only indicates that and recalls it. It may be said indeed to represent the atomic number, since it stands in place of it; but it is made to do so only in order that we may for the time forget this number and have in mind the integral character of the atom. It is not the 4006 parts of sodium hydroxide and 8097 parts of hydrobromic acid, or approximately twice as much of the latter as of the former; it is not these gravimetrically expressed interacting quantities that we are to think of when the formulæ NaOH and HBr are before us, as we too often strive to do; it is not these from a chemical point of view, meaningless numbers of parts, but quantities which are equal in the sense of chemistry, that are expressed as such by these symbolic formulæ. The real purpose of chemical formulation is not to abbreviate or replace language, but to facilitate, if not ensure, abstraction from and non-contemplation of gravimetric numbers.

I have just passed from atomic symbols to the formulæ of molecules; but this was not without warrant. In the form in which I have enunciated the atomic theory, it relates to the chemical interaction of substances, whether compound or simple, and the equality of the quantities concerned is the equality of molecules, since these are the quantities of substances entering into or coming out

from single chemical interactions. Were it not, therefore, for fear of confounding it with the mechanical theory of that name, the atomic theory should be called the molecular theory of chemistry. It might, indeed, have happened to be so called by its author, for Dalton has told us that he had in mind both atom and molecule as names for his chemically ultimate particles, and chose the former because it carried with it the notion of indivisibility. He extended also, as we do, the use of the term "atom" to chemically compound substances, since their combining quantities are chemically indivisible.

Next, I would point out that in the atomic theory the notions of indivisibility and equality are inseparably involved. The indivisibility of atom and molecule is not absolute or ultimate, and Dalton distinctly guarded himself against being understood to claim for the atom more than chemical indivisibility, and chemists of to-day assert no more than this. This indivisibility being conditioned by the equality of molecules, the importance of emphasising it rests only upon the danger, when it is overlooked, of losing sight also of the chemical equality through the gravimetric inequality receiving numerical expression, and thereby conveying the notion of divisibility, though only gravimetrically. The idea of indivisibility in connection with the atom or molecule is intrinsically quite subordinate to that of equality; for equality, being unity or oneness brought into relation with itself, the conception of it carries with it and includes that of indivisibility. Any rational hypothesis as to substances consisting of ultimate particles will include the notion of their being indivisible particles; and the import of the hypothesis in chemical theory must lie, therefore, not in this indivisibility, but in the nature of the equality of the particles. By his atomic theory Dalton asserted that where the substances are different this equality is chemical instead of gravimetric.

Molecules are equal in the sense that they are quantities of their substances which are interdependent and co-ordinate in any and every single chemical change in which they take part together. It is a form of equality for which no close parallel can be found; but as to that it should be remembered that this equality relates to the phenomena of the transformations of substances into each other, which, though they form so large a part of the phenomena of the universe, are fundamentally distinct in nature from the rest of the behaviour of bodies throughout which the substance remains what it was. In some agreement with it there is that of mechanical pressures when these balance or neutralise each other, and therefore are opposite and mutually destructive though equal. But such pressures when exerted in the same direction are also equal in their effect on any body in their path, whereas in chemical interactions the effects of molecules or equal quantities of two unlike substances are only equal in the sense that each is that quantity which interacts with the same quantity of some third substance, which itself proves to be also a chemically equal quantity to them. For the products of the interaction in the one case are in part at least not the same as those in the other, though all prove chemically equal in further interactions.

To give an example, the molecule of ammonia is equal to that of aldehyde in that it combines with it and with it disappears, or ceases to exist as such. For the same reason it is equal to the molecule of hydrocyanic acid, and molecules of aldehyde and hydrocyanic acid equal to each other, because they, too, combine and disappear as such in doing so. But the molecule of ammonia again equals that of aldehyde in effecting transformation of hydrocyanic acid and its own self into something else. And lastly, chemically equal or molecular are the products of these combinations; aldehyde ammonia, ammonium cyanide, and aldehyde-cyanhydrine, not only among themselves, but also with the quantities of ammonia, aldehyde, and hydrocyanic acid from which they come and into which they return in other chemical changes. But with all this quantitative equality in trans-

forming power, the substances produced are unlike and, each to each, peculiar to one of the three acts of chemical combination; and on this account exception may be taken to the treatment of molecules as equal chemical quantities. Yet the equality of molecules here asserted is but an extension of what is meant by the equivalence of certain atoms and radicals, since the atom and the radical are, nowadays, conceptions entirely dependent upon and derived from that of the molecule (apart, of course, from the atomic hypothesis); and this universally allowed equivalence admittedly does not extend to the identity of the products of the replacing activity of the atoms and radicals.

Quantitative equality and equivalency, it is true, have not the same meaning, equivalence being used to denote qualified equality, equality in certain specified ways, of quantities not equal in all other ways and possibly in no other. Quantities of different substances cannot, strictly speaking, ever be equal, and can only be styled so in the sense of being equivalent; for were they equal in every way the substances would obviously be the same. But this fact, if it ever strikes one, is ignored by universal custom, and quantities of substances, however unlike—feathers, air, water, salt, and what not—are taken to be all equal, even by chemists as by the world at large, if only they have the same weight, notwithstanding the incongruities of the substances. I proceed now to show the baselessness of this conviction, but only to bring out more strongly the claim of chemical activity to equal rights with weight or mass in determining what are equal quantities of substances, for I am aware that here I have nothing to tell you that you do not already know. Weight being only the gravitational measure of mass, which itself is independent of it, quantities of substances are held to be equal, when their masses are equal. Now, mass is quantity of matter. But what then is meant by matter? The answer must be either that it is a general term for any and all substances, or else that it is the common basis of all substances, which presents itself in all the different forms which are known to us as such, by virtue of a corresponding variety in its intestinal motions. I gladly pass over the latter answer without discussing it, on the ground that it introduces the subject of the intimate constitution of substances, which it is my set purpose to keep independent of in this discourse. I will only say of it that it would probably be the answer of many physicists and chemists, and yet that it gives such a limitation to the nature of matter as makes the common expression "constitution of matter" devoid of all meaning. That expression means, and can only mean, the constitution of substances in common; and this brings me to the first answer, that matter is the term standing for all substances in common. Now, one thing which all substances possess in common is the property of resisting pressures; pressures not only of moving bodies, but of the motions of the ether and electrons. Measured or quantified, resistance becomes mass, all that can be signified by this term being the quantity of the resistance or inertia a substance exhibits when tested. It is the measure of a property of the substance, that is all; and there is no other way of quantifying a substance than through some one of its properties. No quantities of different substances can, as such, be commensurable throughout; and when compared and measured through some common property, such as the possession of mass, the equivalence or pseudo-equality found by this means is not the same as that found when some other common property is taken as the means of measurement. But experience has shown that though there are several rational and comprehensive ways of instituting, through some common property, comparisons between quantities of different substances, they all, with the exception of that of weighing, agree more or less exactly in pointing to the same order of equivalence, that of chemical activity; for with this are colligated those of gaseous volume and the other well-known physical activities, which give nearly the same quantities

as it gives of different substances as being molecularly equivalent. There are, therefore, essentially only two measures of quantitative equivalency or pseudo-equality between substances, the dynamical and the chemical or molecular, the one wholly independent of and the other wholly dependent upon the particular nature of the substances compared. The former is the measure of dynamical phenomena, those of changes of bodies, due to their impacts and pressures, which may lead to their deformation and disruption, but do not involve transformations of the substances of the bodies into others; the latter is the measure of chemical phenomena, those of changes of bodies induced by such of their interactions as do involve transformations of the substances of the interacting bodies into other substances. Since it is already settled for us by custom that quantities of different substances are to be called equal when or because they are equivalent gravimetrically, and as it is not to be supposed that we shall ever give up calling 16 kilos. of oxygen, of salt, of chalk, and of every other substance, however unlike, equal quantities of them from the gravimetric point of view, we have no choice but also to call molecular quantities of these substances equal from the chemical point of view, if the claim to co-ordination in equality of chemical with gravimetric equivalency is to be asserted and maintained.

(To be continued).

ATOMIC WEIGHT STANDARDS AND PROUT'S HYPOTHESIS.

(PRELIMINARY NOTICE).

By CECIL HOLLINS.

THE reason for the failure of all attempts to express the atomic weights as integers probably lies in the fact that the units chosen so far have not been suitable ones.

Hydrogen was first adopted as the standard because it was the lightest element known. Owing to the difficulty of making direct determinations with this element, an oxygen standard seemed more desirable; accordingly the atomic weight of O was raised from 15.88 to 16.00, thus lowering the unit to 0.9925 of the atomic weight of H. In this scale the atomic weights of the elements exhibit a considerable tendency to agree with Prout's whole-number hypothesis. This is shown in Table I.

TABLE I.

	H=1.	O=16.
Number of integral atomic weights..	6	18
Sum of deviations from integers ..	15.861	10.298

So, by further lowering the unit, we might expect that the atomic weights of the elements would still more nearly approach to integers. Of course, it was useless to attempt to find the G.C.M., but by dividing each atomic weight into the nearest whole number, factors were obtained varying between 1.0101 for He and 0.9859 for Si, the mean being 0.99877. If this be taken as the unit, the atomic weights of the elements show a further tendency to become integral; and, by treating these in the same way, the total deviation is again reduced (the mean factor now = 0.99867). The results for seventeen elements upon whose atomic weights most authorities agree, are given in Table II.

I am engaged in further investigation of the subject, but even as the table stands there is some encouragement shown; for the probability that the total deviation in the last column should not be greater than 0.729 is only 1 in 4×10^{14} ; and even when the elements of atomic weight less than 27 are eliminated, the chances are still 1000 to 1 against the total deviation not being greater than it is.

Possibly the approximate G.C.M. of the factors will give better results than their mean, but this I leave for a

TABLE II.
Atomic weights.

	H=1.	O=16.	Factor 0.99877.	Factor 0.99867.
H	1.00	1.008	1.007	1.005
He	3.93	3.96	3.955	3.950
Li	6.97	7.03	7.021	7.011
Na	22.88	23.05	23.022	22.991
K	38.82	39.11	39.062	39.010
O	15.88	16.00	15.980	15.960
S	31.83	32.07	32.030	31.997
Se	78.6	79.2	79.102	78.997
N	13.93	14.04	14.023	14.004
F	18.91	19.05	19.026	19.002
Cl	35.18	35.45	35.406	35.359
C	11.9	12.0	11.985	11.969
Cr	51.7	52.1	52.036	51.967
Be	9.0	9.1	9.089	9.077
Ca	39.8	40.1	40.080	40.027
Al	26.9	27.1	27.066	27.030
B	10.9	11.0	10.986	10.971
Number of integral atomic weights* ..	3	6	10	15
Total deviation from integers	2.87	1.448	1.064	0.729

* Correct to one decimal place.

further paper, in which I hope to bring the atomic weights of all the elements as near to integers as the seventeen enumerated above.

NOTE ON CEMENT ANALYSIS.

By R. F. YOUNG and B. F. BAKER.

It is often desirable in cement analysis to estimate only CaO. The usual method of procedure is to dissolve the cement in dilute HCl, add excess of AmOH to precipitate silica, Al_2O_3 , and Fe_2O_3 , precipitate the calcium as oxalate, and ignite to CaO.

This method has been found by the authors to give an entirely erroneous result, which may be as much as 1.5 per cent too high or too low.

The reason of this, in our opinion, is that when cement is dissolved in dilute HCl, part of the calcium silicate is not decomposed, but merely dissolves and is precipitated on neutralising the acid with ammonia. This causes a low result; but, on the other hand, the ignited CaO will invariably contain very appreciable quantities of SiO_2 , Al_2O_3 , and Fe_2O_3 , which will cause a high result.

The two following methods have been found to give very satisfactory results:—

I. One grm. of the cement is treated in a conical beaker with a small quantity of concentrated HCl. A little HNO_3 is added, and the contents evaporated to dryness on a sand-bath, and heated till the contents are distinctly red; then treated with dilute HCl, and excess of ammonia added. The SiO_2 , $Al_2(OH)_3$, and $Fe_2(OH)_3$ are filtered off, and the calcium precipitated as oxalate and ignited to CaO in the usual way.

II. One grm. of cement is dissolved in dilute HCl, AmOH added, and the precipitate of iron, alumina, and silica filtered off. This precipitate is re-dissolved in concentrated HCl and re-precipitated by AmOH. The two filtrates and washings are collected, and the calcium estimated as above.

In accurate estimations, it is advisable in both methods to dissolve the ignited CaO after weighing in HCl, and estimate the SiO_2 , Al_2O_3 , and Fe_2O_3 it may contain.

We may mention that we have tried the volumetric estimation of calcium by titrating the excess of a known quantity of standard oxalic acid left after precipitating in ammoniacal solution, and have found it invariably gives

low results. This seems to point to the fact that all the calcium is not precipitated as oxalate.

The Laboratory,
Wouldham Cement Co. (1900),
West Thurrock, Essex.

ON THE PRESENCE OF LIME AS DOLOMITE
IN CERTAIN CULTIVATED SOILS.

By Dr. T. L. PHIPSON.

In a recent examination of certain cane soils from Argentina, yielding only from 8 to 20 tons of cane to the acre, I have met with a singular result, which may partly account for this deficiency, and does not appear to have ever been noticed before.

These soils, of which I have made the complete analysis of eight specimens taken with great care at a depth of 1 foot from the surface, have as their basis a micaceous sand, with little clay, a good amount of nitrogenous humus, and all the physical and chemical elements of fertility; but the lime, which amounts to no less than 1 per cent, appears not to have been affected after ten or twelve years' constant cultivation and cropping. Here are the figures for lime and magnesia:—

	Lime.	Magnesia.
1. Cultivated ten years. Yield 15 tons ..	1.12	1.08
2. Same soil, uncultivated	1.12	1.09
3. Another plantation, same district. Cul- tivated twelve years. Yield, 20 tons ..	1.10	1.08
4. The same from an uncultivated region ..	1.11	1.09
5. Another plantation, cultivated several years. Yield, 8 tons	1.12	1.02
6. Same land, uncultivated	0.95	0.80

It is evident from these results that the lime is present as *dolomite*, which is not readily assimilated.

The phosphoric acid in these soils amounts to about 0.08 per cent, and the alkalis to about 0.12; whilst the humus averages 4 per cent, and the insoluble rock (sand, with golden mica passing into white mica, and a little clay) 70 to 84 per cent.

CHEMICAL NOTICES FROM FOREIGN
SOURCES.

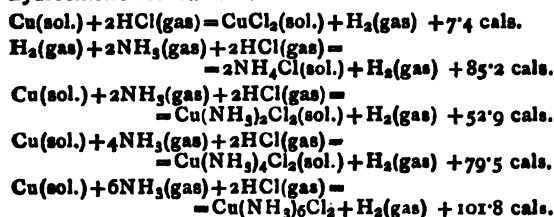
NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Seances, de l'Académie des Sciences. Vol. cxxv., No. 5, August 4, 1902.

Direct Hydrogenation of the Oxides of Nitrogen by the Method of Contact.—Paul Sabatier and J. B. Senderens.—It has been known for some time that finely divided platinum will provoke direct reduction of several nitrogen oxides by means of hydrogen. The authors now perform a series of experiments on the action of reduced nickel and copper in order to investigate a general method of hydrogenation for the volatile organic compounds, and particularly nitrogen derivatives. They find the action of reduced nickel and copper the same as that of spongy platinum.

Gentibiose. Preparation and Properties of Crystalline Gentibiose.—Em. Bourquelot and H. Hérissay.—Previous researches established the fact that gentianose is a hexatriose, $C_{12}H_{22}O_{11}$, which, when treated with invertine or very dilute boiling sulphuric acid, is decomposed, giving one molecule of levulose and one molecule of a hexobiase, which the authors called gentibiose. This latter substance they now investigate more fully. They find the formula, instead of being $C_{12}H_{22}O_{11}$, to be $C_{12}H_{22}O_{11} + 2(CH_4O)$; that is to say, the crystals contain 2 mols. of methyl alcohol. The substance can, however, be obtained in an anhydrous state free from alcohol.

Anhydrous Ammoniacal Cupric Chlorides. Cupro-ammoniac Radicles.—M. Bouzat.—Different cupric salts, such as chlorides, sulphates, acetates, &c., evolve equal quantities of heat on combining with ammonia. This relation points to the existence of complex radicles forming cupro-ammoniac salts. The author now further examines the action of ammonia gas on anhydrous copper salts (chlorides and sulphates). He finds the heat of fixation of the two first molecules of ammonia on to the copper chloride is 45.5 cal., of the next two is 26.6 cal., and of the last two 22.3 cal. The following equations show for each cupro-ammoniac radicle the heat of formation and the heat of substitution of the hydrogen in the hydrochloric acid radicle :—



Action of Nitrous Acid in Alkaline Solution on the α -Substituted β -Ketonic Ethers.—M. Bouveault and René Locquin.—The mechanism of the action of nitrous acid on the α -substituted β -ketonic ethers can be stated as follows :—If the reaction takes place under such conditions that the ether group is not saponified, or if it is saponified in acid solution, it forms an acid and a substituted oxime of glyoxylic ether. If, during the reaction, the ether group is saponified in such a manner as to give the salt $\text{R}-\text{CO}-\text{CH} < \text{R}'^{\text{CO}}_{\text{M}}$, a monoxime of α -diketone and carbonic acid are obtained.

No. 6, August 11.

This number contains no chemical matter.

MISCELLANEOUS.

British Association for the Advancement of Science.—The following are the names of the Officers and Committee of Section B (Chemistry) at the Belfast Meeting of the British Association :—

President—Prof. Edward Divers, M.D., F.R.S., V.P.C.S.
Vice-Presidents—Prof. P. F. Frankland, F.R.S.; Prof. E. A. Letts, D.Sc., Ph.D.; Prof. W. A. Shenstone, F.R.S.
Secretaries—R. F. Blake; M. O. Forster, D.Sc., Ph.D.; Prof. G. G. Henderson, M.A., D.Sc.; Prof. W. J. Pope, F.R.S. (Recorder).

Committee—W. E. Adeney, D.Sc.; E. F. Armstrong, Ph.D.; G. T. Beilby; C. H. Bothamley; Prof. A. Crum Brown, F.R.S.; Sir C. Cameron, M.D.; D. L. Chapman; Prof. A. E. Dixon, M.D.; F. G. Donnan, D.Sc.; T. Fairley; Prof. J. H. Gladstone, F.R.S.; G. Gladstone; R. T. Glazebrook, D.Sc., F.R.S.; Prof. W. D. Halliburton, F.R.S.; Prof. W. N. Hartley, F.R.S.; P. J. Hartog, B.Sc.; W. J. Heath; A. Hutchinson, M.A., Ph.D.; Francis Jones; Prof. E. Knoevenagel; R. N. Lennox; H. Marshall, D.Sc.; W. S. Mills, M.A.; G. T. Morgan, D.Sc.; H. Ramage, B.A.; Lord Rayleigh, F.R.S.; Sir W. C. Roberts-Austen, K.C.B., F.R.S.; Prof. Senior; J. Smyth; Prof. J. J. Sudborough, D.Sc., Ph.D.; W. Thomson; M. W. Travers, D.Sc.; Prof. D. Vorländer; Prof. J. Wertheimer.

The Papers brought before the Section were as follows :—

President's Address—The Atomic Theory without Hypothesis.

Prof. E. A. Letts and W. Caldwell—On Experiments to ascertain the amount of Carbonic Anhydride absorbed from Sea-water by Air.

Prof. E. A. Letts—On the Corrosion of Copper by Sea-water, and on the Detection of Traces of Impurity in the Commercial Metal.

Prof. F. Clowes, D.Sc.—Action of Distilled Water on Lead.

Dr. A. W. Crossley—Hydro-aromatic Compounds with Single Nucleus.

B. Blount—The Undesirability of establishing Standard Analytical Methods.

Dr. C. E. Fawsitt—The Decomposition of Urea.

Prof. W. N. Hartley—Report of Committee on Absorption Spectra.

Dr. E. F. Armstrong—Recent Synthetical Researches in the Glucoside Group.

Dr. E. F. Armstrong—The Synthetical Action of Enzymes.

W. Ackroyd—The Telluric Distribution of the Elements in relation to their Atomic Weights.

Dr. G. T. Morgan—Our present Knowledge of Aromatic Diazo-compounds.

Miss Ida Smedley—The Colour of Iodine-containing Compounds.

J. Hawthorne—Note on a Fourth Methylmorphimethine.

Prof. T. Purdie and Dr. Irvine—The Alkylation of the Sugars.

Prof. G. G. Henderson—Report of the Committee on preparing Statistics concerning the Training of Chemists employed in English Chemical Industries.

Report of the Committee on Isomeric Naphthalene Derivatives.

Report of the Committee on Isomorphous Sulphonic Derivatives of Benzene.

Major W. E. Edwards, Capt. C. H. Living, and Prof. W. R. Hodgkinson—The Reduction of some Metallic Chlorides by Calcium Carbide.

Dr. J. H. Gladstone and W. Hibbert—On Zirconium Hydrate and other Colloids from Elements of the Fourth Group.

Dr. J. H. Gladstone—On Fluorescent and Phosphorescent Diamonds.

Prof. J. J. Sudborough and W. A. Bone—Acid Esters of Methylsuccinic Acids.

H. Hibbert and Prof. J. J. Sudborough—Compounds of Trinitrobenzenes and Alkylated Naphthylamines.

Prof. J. J. Sudborough and K. J. Thompson—Action of Alkalis on Cinnamic Acid Dibromide and its Esters.

Prof. E. A. Letts and J. S. Totton—On the Absorption of Ammonia from Water by Algae.

Prof. E. A. Letts—On Determinations of Atmospheric Carbonic Anhydride made on board the *Discovery* on the Voyage to the Cape and thence to New Zealand.

Prof. R. Meldola—A New Method of causing Isomerisation.

Report of the Committee on the Effect of Gases on Metals.

Report of the Committee on the Nature of Alloys.

Report of the Committee on Preparing a New Series of Wave-length Tables of the Spectra of the Elements.

The Chemical Laboratory Fresenius at Wiesbaden.—During the Summer Term, 1902, the Chemical Laboratory Fresenius was attended by forty-nine Students. Of these, twenty nine were from Germany, four from England, three from Russia, three from Luxemburg, two from Holland, one from Austria, one from France, one from Belgium, one from Italy, one from Spain, one from Denmark, one from Norway, and one from the Transvaal. There were three Assistants in the Instruction Laboratory, and twenty-four in the Versuchsstationen (private laboratories). To the body of teachers belong the Directors, Prof. Dr. H. Fresenius, Prof. Dr. W. Fresenius, Prof. Dr. E. Hintz, and also Dr. Med. G. Frank, Dr. W. Lenz, Dr. L. Grünhut, and J. Huber (Architect). The next Winter Term begins on October 15. During the Summer Term, 1902, besides the scientific researches, a great number of analyses were undertaken in the different departments of the Laboratory (Versuchsstationen), on behalf of trade, manufacture, mining, agriculture, and hygiene.

NOTES AND QUERIES.

Uses of Litmus-paper.—It seems to me to be very apathetic on the part of litmus-paper manufacturers that they do not advertise it largely as an absolutely necessary adjunct of every householder for use in ascertaining the degree of fermentation in milk when supplied to them in such an injurious and avoidable condition; also amongst dairymen for protection against avoidable souring, semi-fermented milk being supplied and delivered to them by dairy farmers. Such an advertisement ought to appear largely in the daily newspapers. I am widely advising the needed use of litmus-paper for the purpose amongst householders and dairymen.—A. ROBINSON.

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Principal—HERBERT TOMLINSON, B.A., F.R.S.

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Organic Chemistry L. GRÜNHUT, Ph.D.
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Chemistry and Analysis of Foods Prof. H. FRESENIUS, Ph.D.
Prof. W. FRESENIUS, Ph.D.
and Prof. E. HINTZ, Ph.D.

Hygiene Dr. med. G. FRANK.
Practical exercises in Bacteriology
Technical Drawing, with exercises .. J. HUBER.

The next Session commences on the 15th of October. The Regulations of the Laboratory and the Syllabus of Lectures will be forwarded gratis on application to C. W. KRIEDEL's Verlag, at Wiesbaden, or to one of the Directors.

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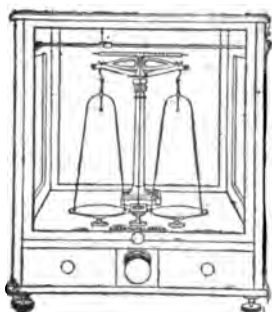
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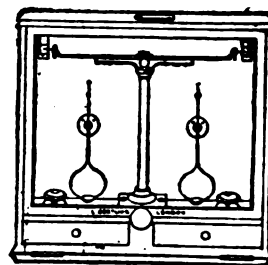


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INAUGURAL ADDRESS OF THE PRESIDENT,
PROF. JAMES DEWAR, M.A., LL.D., D.Sc., F.R.S.

(Concluded from p. 144).

Liquid Hydrogen and Helium.

To the physicist the copious production of liquid air by the methods described was of peculiar interest and value as affording the means of attacking the far more difficult problem of the liquefaction of hydrogen, and even as encouraging the hope that liquid hydrogen might in time be employed for the liquefaction of yet more volatile elements, apart from the importance which its liquefaction must hold in the process of the steady advance towards the absolute zero. Hydrogen is an element of especial interest, because the study of its properties and chemical relations led great chemists like Faraday, Dumas, Daniell, Graham, and Andrews to entertain the view that if it could ever be brought into the state of liquid or solid it would reveal metallic characters. Looking to the special chemical relations of the combined hydrogen in water, alkaline oxides, acids, and salts, together with the behaviour of these substances on electrolysis, we are forced to conclude that hydrogen behaves as the analogue of a metal. After the beautiful discovery of Graham, that palladium can absorb some hundreds of times its own volume of hydrogen, and still retain its lustre and general metallic character, the impression that hydrogen was probably a member of the metallic group became very general. The only chemist who adopted another view was my distinguished predecessor, Professor Odling. In his "Manual of Chemistry," published in 1861, he pointed out that hydrogen has chlorous as well as basic relations, and that they are as decided, important, and frequent as its other relations. From such considerations he arrived at the conclusion that hydrogen is essentially a neutral or intermediate body, and therefore we should not expect to find liquid or solid hydrogen possess the appearance of a metal. This extraordinary prevision, so characteristic of Odling, was proved to be correct some thirty-seven years after it was made. Another curious anticipation was made by Dumas in a letter addressed to Pi&et, in which he says that the metal most analogous to hydrogen is magnesium, and that probably both elements have the same atomic volume, so that the density of hydrogen, for this reason, would be about the value elicited by subsequent experiments. Later on, in 1872, when Newlands began to arrange the elements in periodic groups, he regarded hydrogen as the lowest member of the chlorine family; but Mendeleeff in his later classification placed hydrogen in the group of the alkaline metals; on the other hand, Dr. Johnstone Stoney classes hydrogen with the alkaline earth metals and magnesium. From this speculative divergency it is clear no definite conclusion could be reached regarding the physical properties of liquid or solid hydrogen, and the only way to arrive at the truth was to prosecute low-temperature research until success attended the efforts to produce its liquefaction. This result I definitely obtained in 1898. The case of liquid hydrogen is, in fact, an excellent illustration of the truth already referred to, that no theoretical forecast, however apparently justified by analogy, can be finally accepted as true until confirmed

by actual experiment. Liquid hydrogen is a colourless transparent body of extraordinary intrinsic interest. It has a clearly defined surface, is easily seen, drops well, in spite of the fact that its surface tension is only the thirty-fifth part of that of water, or about one-fifth that of liquid air, and can be poured easily from vessel to vessel. The liquid does not conduct electricity, and, if anything, is slightly diamagnetic. Compared with an equal volume of liquid air, it requires only one-fifth the quantity of heat for vaporisation: on the other hand, its specific heat is ten times that of liquid air or five times that of water. The coefficient of expansion of the fluid is remarkable, being about ten times that of gas; it is by far the lightest liquid known to exist, its density being only one-fourteenth that of water; the lightest liquid previously known was liquid marsh-gas, which is six times heavier. The only solid which has so small density as to float upon its surface is a piece of pith wood. It is by far the coldest liquid known. At ordinary atmospheric pressure it boils at minus 252.5 degrees or 20.5 degrees absolute. The critical point of the liquid is about 29 degrees absolute, and the critical pressure not more than fifteen atmospheres. The vapour of the hydrogen arising from the liquid has nearly the density of air—that is, it is fourteen times that of the gas at the ordinary temperature. Reduction of the pressure by an air-pump brings down the temperature to minus 258 degrees, when the liquid becomes a solid resembling frozen foam, and this by further exhaustion is cooled to minus 260 degrees, or 13 degrees absolute, which is the lowest steady temperature that has been reached. The solid may also be got in the form of a clear transparent ice, melting at about 15 degrees absolute, under a pressure of 55 m.m., possessing the unique density of one-eleventh that of water. Such cold involves the solidification of every gaseous substance but one that is at present definitely known to the chemist, and so liquid hydrogen introduces the investigator to a world of solid bodies. The contrast between this refrigerating substance and liquid air is most remarkable. On the removal of the loose plug of cotton-wool used to cover the mouth of the vacuum vessel in which it is stored, the action is followed by a miniature snowstorm of solid air, formed by the freezing of the atmosphere at the point where it comes into contact with the cold vapour rising from the liquid. This solid air falls into the vessel and accumulates as a white snow at the bottom of the liquid hydrogen. When the outside of an ordinary test-tube is cooled by immersion in the liquid, it is soon observed to fill up with solid air, and if the tube be now lifted out a double effect is visible, for liquid air is produced both in the inside and on the outside of the tube—in the one case by the melting of the solid, and in the other by condensation from the atmosphere. A tuft of cotton-wool soaked in the liquid and then held near the pole of a strong magnet is attracted, and it might be inferred therefrom that liquid hydrogen is a magnetic body. This, however, is not the case: the attraction is due neither to the cotton-wool nor to the hydrogen—which indeed evaporates almost as soon as the tuft is taken out of the liquid—but to the oxygen of the air, which is well known to be a magnetic body, frozen in the wool by the extreme cold.

The strong condensing powers of liquid hydrogen afford a simple means of producing vacua of very high tenuity. When one end of a sealed tube containing ordinary air is placed for a short time in the liquid, the contained air accumulates as a solid at the bottom, while the higher part is almost entirely deprived of particles of gas. So perfect is the vacuum thus formed, that the electric discharge can be made to pass only with the greatest difficulty. Another important application of liquid air, liquid hydrogen, &c., is as analytic agents. Thus, if a gaseous mixture be cooled by means of liquid oxygen, only those constituents will be left in the gaseous state which are less condensable than oxygen. Similarly, if this gaseous residue be in its turn cooled in liquid hydrogen a still further separation will be effected, everything that is less

volatile than hydrogen being condensed to a liquid or solid. By proceeding in this fashion it has been found possible to isolate helium from a mixture in which it is present to the extent of only one part in one thousand. By the evaporation of solid hydrogen under the air-pump we can reach within 13 or 14 degrees of the zero, but there or thereabouts our progress is barred. This gap of 13 degrees might seem at first sight insignificant in comparison with the hundreds that have already been conquered. But to win one degree low down the scale is quite a different matter from doing so at higher temperatures; in fact, to annihilate these few remaining degrees would be a far greater achievement than any so far accomplished in low temperature research. For the difficulty is twofold, having to do partly with process and partly with material. The application of the methods used in the liquefaction of gases becomes continually harder and more troublesome as the working temperature is reduced; thus, to pass from liquid air to liquid hydrogen—a difference of 60 degrees—is, from a thermodynamic point of view, as difficult as to bridge the gap of 150 degrees that separates liquid chlorine and liquid air. By the use of a new liquid gas exceeding hydrogen in volatility to the same extent as hydrogen does nitrogen, the investigator might get to within five degrees of the zero; but even a second hypothetical substance, again exceeding the first one in volatility to an equal extent, would not suffice to bring him quite to the point of his ambition. That the zero will ever be reached by man is extremely improbable. A thermometer introduced into regions outside the uttermost confines of the earth's atmosphere might approach the absolute zero, provided that its parts were highly transparent to all kinds of radiation, otherwise it would be affected by the radiation of the sun, and would therefore become heated. But supposing all difficulties to be overcome, and the experimenter to be able to reach within a few degrees of the zero, it is by no means certain that he would find the near approach of the death of matter sometimes pictured. Any forecast of the phenomena that would be seen must be based on the assumption that there is continuity between the processes studied at attainable temperatures and those which take place at still lower ones. Is such an assumption justified? It is true that many changes in the properties of substances have been found to vary steadily with the degree of cold to which they are exposed. But it would be rash to take for granted that the changes which have been traced in explored regions continue to the same extent and in the same direction in those which are as yet unexplored. Of such a breakdown low-temperature research has already yielded a direct proof at least in one case. A series of experiments with pure metals showed that their electrical resistance gradually decreases as they are cooled to lower and lower temperatures, in such ratio that it appeared probable that at the zero of absolute temperature they would have no resistance at all and would become perfect conductors of electricity. This was the inference that seemed justifiable by observations taken at depths of cold which can be obtained by means of liquid air and less powerful refrigerants. But with the advent of the more powerful refrigerant liquid hydrogen it became necessary to revise that conclusion. A discrepancy was first observed when a platinum resistance thermometer was used to ascertain the temperature of that liquid boiling under atmospheric and reduced pressure. All known liquids, when forced to evaporate quickly by being placed in the exhausted receiver of an air-pump, undergo a reduction in temperature, but when hydrogen was treated in this way it appeared to be an exception. The resistance thermometer showed no such reduction as was expected, and it became a question whether it was the hydrogen or the thermometer that was behaving abnormally. Ultimately, by the adoption of other thermometrical appliances, the temperature of the hydrogen was proved to be lowered by exhaustion as theory indicated. Hence it was the platinum thermometer which had broken down: in other words,

the electrical resistance of the metal employed in its construction was not, at temperatures about minus 250° C., decreased by cold in the same proportion as at temperatures about minus 200°. This being the case, there is no longer any reason to suppose that at the absolute zero platinum would become a perfect conductor of electricity; and in view of the similarity between the behaviour of platinum and that of other pure metals in respect of temperature and conductivity, the presumption is that the same is true of them also. At any rate, the knowledge that in the case of at least one property of matter we have succeeded in attaining a depth of cold sufficient to bring about unexpected change in the law expressing the variation of that property with temperature, is sufficient to show the necessity for extreme caution in extending our inferences regarding the properties of matter near the zero of temperature. Lord Kelvin evidently anticipates the possibility of more remarkable electrical properties being met with in the metals near the zero. A theoretical investigation on the relation of "electrons" and atoms has led him to suggest a hypothetical metal having the following remarkable properties:—Below 1 degree absolute it is a perfect insulator of electricity, at 2 degrees it shows noticeable conductivity, and at 6 degrees it possesses high conductivity. It may safely be predicted that liquid hydrogen will be the means by which many obscure problems of physics and chemistry will ultimately be solved, so that the liquefaction of the last of the old permanent gases is as pregnant now with future consequences of great scientific moment as was the liquefaction of chlorine in the early years of the last century.

The next step towards the absolute zero is to find another gas more volatile than hydrogen, and that we possess in the gas occurring in cleveite, identified by Ramsay as helium, a gas which is widely distributed, like hydrogen, in the sun, stars, and nebulae. A specimen of this gas was subjected by Olszewski to liquid air temperatures, combined with compression and subsequent expansion, following the Cailletet method, and resulted in his being unable to discover any appearance of liquefaction, even in the form of mist. His experiments led him to infer that the boiling-point of the substance is probably below 9 degrees absolute. After Lord Rayleigh had found a new source of helium in the gases which are derived from the Bath springs, and liquid hydrogen became available as a cooling agent, a specimen of helium cooled in liquid hydrogen showed the formation of fluid, but this turned out to be owing to the presence of an unknown admixture of other gases. As a matter of fact, a year before the date of this experiment I had recorded indications of the presence of unknown gases in the spectrum of helium derived from this source. When subsequently such condensable constituents were removed, the purified helium showed no signs of liquefaction, even when compressed to 80 atmospheres, while the tube containing it was surrounded with solid hydrogen. Further, on suddenly expanding, no instantaneous mist appeared. Thus helium was definitely proved to be a much more volatile substance than hydrogen in either the liquid or solid condition. The inference to be drawn from the adiabatic expansion effected under the circumstances is that helium must have touched a temperature of from 9 to 10 degrees for a short time without showing any signs of liquefaction, and consequently that the critical-point must be still lower. This would force us to anticipate that the boiling-point of the liquid will be about 5 degrees absolute, or liquid helium will be four times more volatile than liquid hydrogen, just as liquid hydrogen is four times more volatile than liquid air. Although the liquefaction of the gas is a problem for the future, this does not prevent us from safely anticipating some of the properties of the fluid body. It would be twice as dense as liquid hydrogen, with a critical pressure of only 4 or 5 atmospheres. The liquid would possess a very feeble surface tension, and its compressibility and expansibility would be about four times that of liquid hydrogen, while the heat required to vaporise the mole-

cule would be about one-fourth that of liquid hydrogen. Heating the liquid 1 degree above its boiling-point would raise the pressure by $1\frac{1}{2}$ atmospheres, which is more than four times the increment for liquid hydrogen. The liquid would be only seventeen times denser than its vapour, whereas liquid hydrogen is sixty-five times denser than the gas it gives off. Only some 3 or 4 degrees would separate the critical temperature from the boiling-point and the melting-point, whereas in liquid hydrogen the separation is respectively 10 and 15 degrees. As the liquid refractivities for oxygen, nitrogen, and hydrogen are closely proportional to the gaseous values, and as Lord Rayleigh has shown that helium has only one-fourth the refractivity of hydrogen, although it is twice as dense, we must infer that the refractivity of liquid helium would also be about one-fourth that of liquid hydrogen. Now hydrogen has the smallest refractivity of any known liquid, and yet liquid helium will have only about one-fourth of this value—comparable, in fact, with liquid hydrogen just below its critical point. This means that the liquid will be quite exceptional in its optical properties, and very difficult to see. This may be the explanation of why no mist has been seen on its adiabatic expansion from the lowest temperatures. Taking all these remarkable properties of the liquid into consideration, one is afraid to predict that we are at present able to cope with the difficulties involved in its production and collection. Provided the critical-point is, however, not below 8 degrees absolute, then from the knowledge of the conditions that are successful in producing a change of state in hydrogen through the use of liquid air, we may safely predict that helium can be liquefied by following similar methods. If, however, the critical-point is as low as 6 degrees absolute, then it would be almost hopeless to anticipate success by adopting the process that works so well with hydrogen. The present anticipation is that the gas will succumb after being subjected to this process, only, instead of liquid air under exhaustion being used as the primary cooling agent, liquid hydrogen evaporated under similar circumstances must be employed. In this case the resulting liquid would require to be collected in a vacuum vessel, the outer walls of which are immersed in liquid hydrogen. The practical difficulties and the cost of the operation will be very great; but, on the other hand, the descent to a temperature within 5 degrees of the zero would open out new vistas of scientific inquiry, which would add immensely to our knowledge of the properties of matter. To command in our laboratories a temperature which would be equivalent to that which a comet might reach at an infinite distance from the sun would indeed be a great triumph for science. If the present Royal Institution attack on helium should fail, then we must ultimately succeed by adopting a process based on the mechanical production of cold through the performance of external work. When a turbine can be worked by compressed helium, the whole of the mechanism and circuits being kept surrounded with liquid hydrogen, then we need hardly doubt that the liquefaction will be effected. In all probability gases other than helium will be discovered of greater volatility than hydrogen. It was at the British Association meeting in 1896 that I made the first suggestion of the probable existence of an unknown element which would be found to fill up the gap between argon and helium, and this anticipation was soon taken up by others and ultimately confirmed. Later, in the Bakerian Lecture for 1901, I was led to infer that another member of the helium group might exist having the atomic weight about 2, and this would give us a gas still more volatile, with which the absolute zero might be still more nearly approached. It is to be hoped that some such element or elements may yet be isolated and identified as coronium or nebulium. If amongst the unknown gases possessing a very low critical-point some have a high critical pressure, instead of a low one, which ordinary experience would lead us to anticipate, then such difficulty liquefiable gases would produce fluids having different physical properties

from any of those with which we are acquainted. Again, gases may exist having smaller atomic weights and densities than hydrogen, yet all such gases must, according to our present views of the gaseous state, be capable of liquefaction before the zero of temperature is reached. The chemists of the future will find ample scope for investigation within the apparently limited range of temperature which separates solid hydrogen from the zero. Indeed, great as is the sentimental interest attached to the liquefaction of these refractory gases, the importance of the achievement lies rather in the fact that it opens out new fields of research and enormously widens the horizon of physical science, enabling the natural philosopher to study the properties and behaviour of matter under entirely novel conditions. This department of enquiry is as yet only in its infancy, but speedy and extensive developments may be looked for, since within recent years several special cryogenic laboratories have been established for the prosecution of such researches, and a liquid-air plant is becoming a common adjunct to the equipment of the ordinary laboratory.

The Upper Air and Auroras.

The present liquid ocean, neglecting everything for the moment but the water, was at a previous period of the earth's history part of the atmosphere, and its condensation has been brought about by the gradual cooling of the earth's surface. This resulting ocean is subjected to the pressure of the remaining uncondensed gases, and as these are slightly soluble they dissolve to some extent in the fluid. The gases in solution can be taken out by distillation or by exhausting the water, and if we compare their volume with the volume of the water as steam, we should find about 1 volume of air in 60,000 volumes of steam. This would then be about the rough proportion of the relatively permanent gas to condensable gas which existed in the case of the vaporised ocean. Now let us assume the surface of the earth gradually cooled to some 200 degrees below the freezing-point; then, after all the present ocean was frozen, and the climate became three times more intense than any arctic frost, a new ocean of liquid air would appear, covering the entire surface of the frozen globe about thirty-five feet deep. We may now apply the same reasoning to the liquid air ocean that we formerly did to the water one, and this would lead us to anticipate that it might contain in solution some gases that may be far less condensable than the chief constituents of the fluid. In order to separate them we must imitate the method of taking the gases out of water. Assume a sample of liquid air cooled to the low temperature that can be reached by its own evaporation, connected by a pipe to a condenser cooled in liquid hydrogen; then any volatile gases present in solution will distil over with the first portions of the air, and can be pumped off, being uncondensable at the temperature of the condenser. In this way, a gas mixture, containing, of the known gases, free hydrogen, helium, and neon, has been separated from liquid air. It is interesting to note in passing that the relative volatilities of water and oxygen are in the same ratio as those of liquid air and hydrogen, so that the analogy between the ocean of water and that of liquid air has another suggestive parallel. The total uncondensable gas separated in this way amounts to about one fifty-thousandth of the volume of the air, which is about the same proportion as the air dissolved in water. That free hydrogen exists in air in small amount is conclusively proved, but the actual proportion found by the process is very much smaller than Gautier has estimated by the combustion method. The recent experiments of Lord Rayleigh show that Gautier, who estimated the hydrogen present as one five-thousandth, has in some way produced more hydrogen than he can manage to extract from pure air by a repetition of the same process. The spectroscopic examination of these gases throws new light upon the question of the aurora and the nature of the upper air. On passing electric discharges through the

tubes containing the most volatile of the atmospheric gases, they glow with a bright orange light, which is especially marked at the negative pole. The spectroscopic shows that this light consists, in the visible part of the spectrum, chiefly of a succession of strong rays in the red, orange, and yellow, attributed to hydrogen, helium, and neon. Besides these, a vast number of rays, generally less brilliant, are distributed through the whole length of the visible spectrum. The greater part of these rays are of, as yet, unknown origin. The violet and ultra-violet part of the spectrum rivals in strength that of the red and yellow rays. As these gases probably include some of the gases that pervade interplanetary space, search was made for the prominent nebular, coronal, and auroral lines. No definite lines agreeing with the nebular spectrum could be found, but many lines occurred closely coincident with the coronal and auroral spectrum. But before discussing the spectroscopic problem it will be necessary to consider the nature and condition of the upper air.

According to the old law of Dalton, supported by the modern dynamical theory of gases, each constituent of the atmosphere while acted upon by the force of gravity forms a separate atmosphere, completely independent, except as to temperature, of the others, and the relations between the common temperature and the pressure and altitude for each specific atmosphere can be definitely expressed. If we assume the altitude and temperature known, then the pressure can be ascertained for the same height in the case of each of the gaseous constituents, and in this way the percentage composition of the atmosphere at that place may be deduced. Suppose we start with a surface atmosphere having the composition of our air, only containing two ten-thousandths of hydrogen, then at thirty-seven miles, if a sample could be procured for analysis, we believe that it would be found to contain 12 per cent of hydrogen and only 10 per cent of oxygen. The carbonic acid practically disappears; and by the time we reach forty-seven miles, where the temperature is minus 132 degrees, assuming a gradient of 3.2 degrees per mile, the nitrogen and oxygen have so thinned out that the only constituent of the upper air which is left is hydrogen. If the gradient of temperature were doubled, the elimination of the nitrogen and oxygen would take place by the time thirty-seven miles was reached, with a temperature of minus 220 degrees. The permanence of the composition of the air at the highest altitudes, as deduced from the basis of the dynamical theory of gases, has been discussed by Stoney, Bryan, and others. It would appear that there is a consensus of opinion that the rate at which gases like hydrogen and helium could escape from the earth's atmosphere would be excessively slow. Considering that to compensate any such loss the same gases are being supplied by actions taking place in the crust of the earth, we may safely regard them as necessarily permanent constituents of the upper air. The temperature at the elevations we have been discussing would not be sufficient to cause any liquefaction of the nitrogen and oxygen, the pressure being so low. If we assume the mean temperature as about the boiling-point of oxygen at atmospheric pressure, then a considerable amount of the carbonic acid must solidify as a mist, if the air from a lower level be cooled to this temperature; and the same result might take place with other gases of relatively small volatility which occur in air. This would explain the clouds that have been seen at an elevation of fifty miles, without assuming the possibility of water vapour being carried up so high. The temperature of the upper air must be above that on the vapour pressure curve corresponding to the barometric pressure at the locality, otherwise liquid condensation must take place. In other words, the temperature must be above the dew-point of air at that place. At higher elevations, on any reasonable assumption of temperature distribution, we inevitably reach a temperature where the air would condense, just as Fourier and Poisson supposed it would, unless the

temperature is arrested in some way from approaching the zero. Both ultra-violet absorption and the prevalence of electric storms may have something to do with the maintenance of a higher mean temperature. The whole mass of the air above forty miles is not more than one seven-hundredth part of the total mass of the atmosphere, so that any rain or snow of liquid or solid air, if it did occur, would necessarily be of a very tenuous description. In any case, the dense gases tend to accumulate in the lower strata, and the lighter ones to predominate at the higher altitudes, always assuming that a steady state of equilibrium has been reached. It must be observed, however, that a sample of air taken at an elevation of nine miles has shown no difference in composition from that at the ground, whereas, according to our hypothesis, the oxygen ought to have been diminished to 17 per cent, and the carbonic acid should also have become much less. This can only be explained by assuming that a large intermixture of different layers of the atmosphere is still taking place at this elevation. This is confirmed by a study of the motions of clouds about six miles high, which reveals an average velocity of the air currents of some seventy miles an hour; such violent winds must be the means of causing the intermingling of different atmospheric strata. Some clouds, however, during hot and thundery weather, have been seen to reach an elevation of seventeen miles, so that we have direct proof that on occasion the lower layers of atmosphere are carried to a great elevation. The existence of an atmosphere at more than a hundred miles above the surface of the earth is revealed to us by the appearance of meteors and fireballs, and when we can take photographs of the spectrum of such apparitions we shall learn a great deal about the composition of the upper air. In the meantime Pickering's solitary spectrum of a meteor reveals an atmosphere of hydrogen and helium, and so far this is corroborative of the doctrine we have been discussing. It has long been recognised that the aurora is the result of electric discharges within the limits of the earth's atmosphere, but it was difficult to understand why its spectrum should be so entirely different from anything which could be produced artificially by electric discharges through rarefied air at the surface of the earth. Writing in 1879, Rand Capron, after collecting all the recorded observations, was able to enumerate no more than nine auroral rays, of which but one could with any probability be identified with rays emitted by atmospheric air under an electric discharge. Vogel attributed this want of agreement between nature and experiment, in a vague way, to difference of temperature and pressure; and Zollner thought the auroral spectrum to be one of a different order, in the sense in which the line and band spectra of nitrogen are said to be of different orders. Such statements were merely confessions of ignorance. But since that time observations of the spectra of auroras have been greatly multiplied, chiefly through the Swedish and Danish Polar Expeditions, and the length of spectrum recorded on the ultra-violet side has been greatly extended by the use of photography, so that, in a recent discussion of the results, M. Henri Stassano is able to enumerate upwards of one hundred auroral rays, of which the wavelength is more or less approximately known, some of them far in the ultra-violet. Of this large number of rays he is able to identify, within the probable limits of errors of observation, about two-thirds as rays, which Professor Liveing and myself have observed to be emitted by the most volatile gases of atmospheric air unliquefiable at the temperature of liquid hydrogen. Most of the remainder he ascribes to argon, and some he might, with more probability, have identified with krypton or xenon rays, if he had been aware of the publication of wave-lengths of the spectra of those gases, and the identification of one of the highest rays of krypton with that most characteristic of auroras. The rosy tint often seen in auroras, particularly in the streamers, appears to be due mainly to neon, of which the spectrum is remarkably rich in red and orange rays. One or two neon rays are amongst those most frequently

observed, while the red ray of hydrogen and one red ray of krypton have been noticed only once. The predominance of neon is not surprising, seeing that from its relatively greater proportion in air and its low density it must tend to concentrate at higher elevations. So large a number of probable identifications warrants the belief that we may yet be able to reproduce in our laboratories the auroral spectrum in its entirety. It is true that we have still to account for the appearance of some, and the absence of other, rays of the newly discovered gases, which in the way in which we stimulate them appear to be equally brilliant, and for the absence, with one doubtful exception, of all the rays of nitrogen. If we cannot give the reason of this, it is because we do not know the mechanism of luminescence—nor even whether the particles which carry the electricity are themselves luminous, or whether they only produce stresses causing other particles which encounter them to vibrate; yet we are certain that an electric discharge in a highly rarefied mixture of gases lights one element, and not another, in a way which, to our ignorance, seems capricious. The Swedish North Polar Expedition concluded from a great number of trigonometrical measurements that the average above the ground of the base of the aurora was fifty kilometres (thirty-four miles) at Cape Thorsden, Spitzbergen; at this height the pressure of the nitrogen of the atmosphere would be only about one-tenth of a millimetre, and Moissan and Deslandres have found that in atmospheric air, at pressures less than one millimetre the rays of nitrogen and oxygen fade and are replaced by those of argon and by five new rays which Stassano identifies with rays of the more volatile gases measured by us. Also Collie and Ramsay's observations on the distance to which electrical discharges of equal potential traverse different gases explosively throw much light on the question; for they find that, while for helium and neon this distance is from 250 to 300 m.m., for argon it is 45½ m.m., for hydrogen it is 39 m.m., and for air and oxygen still less. This indicates that a good deal depends on the very constitution of the gases themselves, and certainly helps us to understand why neon and argon, which exist in the atmosphere in larger proportions than helium, krypton, or xenon, should make their appearance in the spectrum of auroras almost to the exclusion of nitrogen and oxygen. How much depends not only on the constitution and it may be temperature of the gases, but also on the character of the electric discharge, is evident from the difference between the spectra at the cathode and anode in different gases, notably in nitrogen and argon, and not less remarkably in the more volatile compounds of the atmosphere. Paulsen thinks the auroral spectrum wholly due to cathodic rays. Without stopping to discuss that question, it is certain that changes in the character of the electric discharge produce definite changes in the spectra excited by them. It has long been known that in many spectra the rays which are inconspicuous with an uncondensed electric discharge become very pronounced when a Leyden jar is in the circuit. This used to be ascribed to a higher temperature in this condensed spark, though measurements of that temperatures have not borne out the explanation. Schuster and Hemaalech have shown that these changes of spectra are in part due to the oscillatory character of the condenser discharge which may be enhanced by self-induction, and the corresponding change of spectrum thereby made more pronounced. Lightning we should expect to resemble condensed discharge much more than aurora, but this is not borne out by the spectrum. Pickering's recent analysis of the spectrum of a flash obtained by photography shows, out of nineteen lines measured by him, only two which can be assigned with probability to nitrogen and oxygen, while three hydrogen rays most likely due to water are very conspicuous, and eleven may be reasonably ascribed to argon, krypton, and xenon, one to more volatile gas of the neon class, and the brightest ray of all is but a very little less re-rangible than

the characteristic auroral ray, and coincides with a strong ray of calcium, but also lies between, and close to, an argon and a neon ray, neither of them weak rays. There may be some doubt about the identification of the spectral rays of auroras because of the wide limits of the probable errors in measuring wave-lengths so faint as most of them are, but there is no such doubt about the wave-lengths of the rays in solar protuberances measured by Deslandres and Hale. Stassano found that these rays, forty-four in number, lying between the Fraunhofer line F and 3148 in the ultra-violet, agree very closely with rays which Professor Liveing and myself measured in the spectra of the most volatile atmospheric gases. It will be remembered that one of the earliest suggestions as to the nature of solar prominences was that they were solar auroras. This supposition helped to explain the marvellous rapidity of their changes, and the apparent suspension of brilliant self-luminous clouds at enormous heights above the sun's surface. Now the identification of the rays of their spectra with those of the most volatile gases, which also furnish many of the auroral rays, certainly supports that suggestion. A stronger support, however, seems to be given to it by the results obtained at the total eclipse of May, 1901, by the American expedition to Sumatra. In the *Astrophysical Journal* for June last is a list of 339 lines in the spectrum of the corona photographed by Humphreys, during totality, with a very large concave grating. Of these no fewer than 209 do not differ from lines we have measured in the most volatile gases of the atmosphere, or in krypton or xenon, by more than one unit of wave-length on Armstrong's scale, a quantity within the limit of probable error. Of the remainder, a good many agree to a like degree with argon lines, a very few with oxygen lines, and still fewer with nitrogen lines; the characteristic green auroral ray, which is not in the range of Humphreys' photographs, also agrees within a small fraction of a unit of wave-length with one of the rays emitted by the most volatile atmospheric gas. Taking into account the Fraunhofer lines H, K, and G, usually ascribed to calcium, there remain only fifty-five lines of the 339 unaccounted for to the degree of probability indicated. Of these considerably more than half are very weak lines which have not depicted themselves on more than one of the six films exposed, and extend but a very short distance into the sun's atmosphere. There are, however, seven which are stronger lines, and reach to a considerable height above the sun's rim, and all have depicted themselves on at least four of the six films. If there be no considerable error in the wave-lengths assigned (and such is not likely to be the case), these lines may perhaps be due to some volatile element which may yet be discovered in our atmosphere. However that may be, the very great number of close coincidences between the auroral rays and those which are emitted under electric excitement by gases of our atmosphere almost constrains us to believe, what is indeed most probable on other grounds, that the sun's coronal atmosphere is composed of the same substances as the earth's, and that it is rendered luminous in the same way—namely, by electric discharges. This conclusion has plainly an important bearing on the explanation which should be given of the outburst of new stars and of the extraordinary and rapid changes in their spectra. Moreover, leaving on one side the question whether gases ever become luminous by the direct action of heat, apart from such transfers of energy as occurred in chemical change and electric disturbance, it demands a revision of the theories which attribute more permanent differences between the spectra of different stars to differences of temperature, and a fuller consideration of the question whether they cannot with better reason be explained by differences in the electric conditions which prevail in the stellar atmosphere.

If we turn to the question what is the cause of the electric discharges which are generally believed to occasion auroras, but of which little more has hitherto been known than that they are connected with sun-spots

and solar eruptions, recent studies of electric discharges in high vacuum, with which the names of Crookes, Röntgen, Lenard, and J. J. Thomson will always be associated, have opened the way for Arrhenius to suggest a definite and rational answer. He points out that the frequent disturbances which we know to occur in the sun must cause electric discharges in the sun's atmosphere far exceeding any that occur in that of the earth. These will be attended with an ionisation of the gases, and the negative ions will stream away through the outer atmosphere of the sun into the interplanetary space, becoming, as Wilson has shown, nuclei of aggregation of condensable vapours and cosmic dust. The liquid and solid particles thus formed will be of various sizes; the larger will gravitate back to the sun, while those with diameters less than one and a half thousandths of a millimetre, but nevertheless greater than a wave-length of light, will, in accordance with Clerk-Maxwell's electromagnetic theory, be driven away from the sun by the incidence of the solar rays upon them, with velocities which may become enormous, until they meet other celestial bodies, or increase their dimensions by picking up more cosmic dust or diminish them by evaporation. The earth will catch its share of such particles on the side which is turned towards the sun, and its upper atmosphere will thereby become negatively electrified until the potential of the charge reaches such a point that a discharge occurs, which will be repeated as more charged particles reach the earth. This theory not only accounts for the auroral discharges, and the coincidence of their times of greatest frequency with those of the maxima of sunspots, but also for the minor maxima and minima. The vernal and autumnal maxima occur when the line through the earth and sun has its greatest inclination to the solar equator, so that the earth is more directly exposed to the region of maximum of sun-spots, while the twenty-six days period corresponds closely with the period of rotation of that part of the solar surface where faculae are most abundant. J. J. Thomson has pointed out, as a consequence of the Richardson observations, that negative ions will be constantly streaming from the sun merely regarded as a hot body, but this is not inconsistent with the supposition that there will be an excess of this emission in eruptions, and from the regions of faculae. Arrhenius' theory accounts also, in a way which seems the most satisfactory hitherto enunciated, for the appearances presented by comets. The solid parts of these objects absorb the sun's rays, and as they approach the sun become heated on the side turned towards him until the volatile substances frozen in or upon them are evaporated and diffused in the gaseous state in surrounding space, where they get cooled to the temperature of liquefaction and aggregated in drops about the negative ions. The larger of these drops gravitate towards the sun and form clouds of the coma about the head, while the smaller are driven by the incidence of the sun's light upon them away from the sun and form the tail. The curvature of the tail depends, as Bredichin has shown, on the rate at which the particles are driven, which in turn depends on the size and specific gravity of the particles, and these will vary with the density of the vapour from which they are formed and the frequency of the negative ions which collect them. In any case Arrhenius' theory is a most suggestive one, not only with reference to auroras and comets, and the solar corona and chromosphere, but also as to the constitution of the photosphere itself.

Various Low Temperature Researches.

We may now summarise some of the results which have already been attained by low-temperature studies. In the first place, the great majority of chemical interactions are entirely suspended, but an element of such exceptional powers of combination as fluorine is still active at the temperature of liquid air. Whether solid fluorine and liquid hydrogen would interact no one can at present say.

Bodies naturally become denser, but even a highly expansive substance like ice does not appear to reach the density of water at the lowest temperature. This is confirmatory of the view that the particles of matter under such conditions are not packed in the closest possible way. The force of cohesion is greatly increased at low temperatures, as is shown by the additional stress required to rupture metallic wires. This fact is of interest in connection with two conflicting theories of matter. Lord Kelvin's view is that the forces that hold together the particles of bodies may be accounted for without assuming any other agency than gravitation or any other law than the Newtonian. An opposite view is that the phenomena of the aggregation of molecules depend upon the molecular vibration as a physical cause. Hence, at the zero of absolute temperature, this vibrating energy being in complete abeyance, the phenomena of cohesion should cease to exist, and matter generally be reduced to an incoherent heap of cosmic dust. This second view receives no support from experiment.

The photographic action of light is diminished at the temperature of liquid air to about 20 per cent of its ordinary efficiency, and at the still lower temperature of liquid hydrogen only about 10 per cent of the original sensitivity remains. At the temperature of liquid air or liquid hydrogen a large range of organic bodies and many inorganic ones acquire under exposure to violet light the property of phosphorescence. Such bodies glow faintly so long as they are kept cold, but become exceedingly brilliant during the period when the temperature is rising. Even solid air is a phosphorescent body. All the alkaline earth sulphides which phosphoresce brilliantly at the ordinary temperature lose this property when cooled, to be revived on heating; but such bodies in the first instance may be stimulated through the absorption of light at the lowest temperatures. Radio-active bodies, on the other hand, like radium, which are naturally self-luminous, maintain this luminosity unimpaired at the very lowest temperatures, and are still capable of inducing phosphorescence in bodies like the platino-cyanides. Some crystals become for a time self-luminous when cooled in liquid air or hydrogen, owing to the induced electric stimulation causing discharges between the crystal molecules. This phenomenon is very pronounced with nitrate of uranium and some platino-cyanides.

In conjunction with Professor Fleming, a long series of experiments was made on the electric and magnetic properties of bodies at low temperatures. The subjects that have been under investigation may be classified as follows:—The Thermo-electric Powers of Pure Metals; the Magnetic Properties of Iron and Steel; Dielectric Constants; the Magnetic and Electric Constants of Liquid Oxygen; Magnetic Susceptibility.

The investigations have shown that electric conductivity in pure metals varies almost inversely as the absolute temperature down to *minus* 200 degrees, but that this law is greatly affected by the presence of the most minute amount of impurity. Hence the results amount to a proof that electric resistance in pure metals is closely dependent upon the molecular or atomic motion which gives rise to temperature, and that the process by which the energy constituting what is called an electric current is dissipated essentially depends upon non-homogeneity of structure and upon the absolute temperature of the material. It might be inferred that at the zero of absolute temperature resistance would vanish altogether, and all pure metals become perfect conductors of electricity. This conclusion, however, has been rendered very doubtful by subsequent observations made at still lower temperatures, which appear to point to an ultimate finite resistance. Thus the temperature at which copper was assumed to have no resistance was *minus* 223 degrees, but that metal has been cooled to *minus* 253 degrees without getting rid of all resistance. The reduction in resistance of some of the metals at the boiling-point of hydrogen is very remarkable. Thus copper has only 1 per cent, gold and platinum

3 per cent, and silver 4 per cent of the resistance they possessed at zero C., but iron still retains 12 per cent of its initial resistance. In the case of alloys and impure metals, cold brings about a much smaller decrease in resistivity, and in the case of carbon and insulators like gutta-percha, glass, ebonite, &c., their resistivity steadily increases. The enormous increase in resistance of bismuth when transversely magnetised and cooled was also discovered in the course of these experiments. The study of dielectric constants at low temperatures has resulted in the discovery of some interesting facts. A fundamental deduction from Maxwell's theory is that the square of the refractive index of a body should be the same number as its dielectric constant. So far, however, from this being the case generally, the exceptions are far more numerous than the coincidences. It has been shown in the case of many substances, such as ice and glass, that an increase in the frequency of the alternating electro-motive force results in a reduction of the dielectric constant to a value more consistent with Maxwell's law. By experiments upon many substances it is shown that even a moderate increase of frequency brings the large dielectric constant to values quite near to that required by Maxwell's law. It was thus shown that low temperature has the same effect as high frequency in annulling the abnormal dielectric values. The exact measurement of the dielectric constant of liquid oxygen, as well as its magnetic permeability, combined with the optical determination of the refractive index, showed that liquid oxygen strictly obeys Maxwell's electro-optic law even at very low electric frequencies. In magnetic work the result of greatest value is the proof that magnetic susceptibility varies inversely as the absolute temperature. This shows that the magnetisation of paramagnetic bodies is an affair of orientation of molecules, and it suggests that at the absolute zero all the feebly paramagnetic bodies will be strongly magnetic. The diamagnetism of bismuth was found to be increased at low temperatures. The magnetic moment of a steel magnet is temporarily increased by cooling in liquid air, but the increase seems to have reached a limit, because on further cooling to the temperature of liquid hydrogen hardly any further change was observed. The study of the thermo-electric relations of the metals at low temperatures resulted in a great extension of the well-known Tait Thermo-electric Diagram. Tait found that the thermo-electric power of the metals could be expressed by a linear function of the absolute temperature, but at the extreme range of temperature now under consideration this law was found not to hold generally; and, further, it appeared that many abrupt electric changes take place, which originate probably from specific molecular changes occurring in the metal. The thermo-electric neutral points of certain metals, such as lead and gold, which are located about or below the boiling-point of hydrogen, have been found to be a convenient means of defining specific temperatures in this exceptional part of the scale.

The effect of cold upon the life of living organisms is a matter of great intrinsic interest, as well as of wide theoretical importance. Experiment indicates that moderately high temperatures are much more fatal, at least to the lower forms of life, than are exceedingly low ones. Professor McKendrick froze for an hour at a temperature of -182°C . samples of meat, milk, &c., in sealed tubes; when these were opened after being kept at blood heat for a few days, their contents were found to be quite putrid. More recently, some more elaborate tests were carried out at the Jenner Institute of Preventive Medicine on a series of typical bacteria. These were exposed to the temperature of liquid air for twenty hours, but their vitality was not affected, their functional activities remained unimpaired, and the cultures which they yielded were normal in every respect. The same result was obtained when liquid hydrogen was substituted for air. A similar persistence of life in seeds has been demonstrated even at the lowest temperatures; they were frozen for over a hundred hours

in liquid air, at the instance of Messrs. Brown and Escombe, with no other result than to affect their protoplasm with a certain inertness, from which it recovered with warmth. Subsequently, commercial samples of barley, pea, vegetable-marrow, and mustard seeds were literally steeped for six hours in liquid hydrogen at the Royal Institution, yet when they were sown by Sir W. T. Thiselton Dyer at Kew in the ordinary way, the proportion in which germination occurred was no less than in the other batches of the same seeds which had suffered no abnormal treatment. Bacteria are minute vegetable cells, the standard of measurement for which is the "mikron." Yet it has been found possible to completely triturate these microscopic cells, when the operation is carried out at the temperature of liquid air, the cells then being frozen into hard breakable masses. The typhoid organism has been treated in this way, and the cell plasma obtained for the purpose of studying its toxic and immunising properties. It would hardly have been anticipated that liquid air should find such immediate application in biological research. A research by Professor Macfadyen, just concluded, has shown that many varieties of micro-organisms can be exposed to the temperature of liquid air for a period of six months without any appreciable loss of vitality, although at such a temperature the ordinary chemical processes of the cell must cease. At such a temperature the cells cannot be said to be either alive or dead, in the ordinary acceptation of these words. It is a new and hitherto unobtained condition of living matter—a third state. A final instance of the application of the above methods may be given. Certain species of bacteria during the course of their vital processes are capable of emitting light. If, however, the cells be broken up at the temperature of liquid air, and the crushed contents brought to the ordinary temperature, the luminosity function is found to have disappeared. This points to the luminosity not being due to the action of a ferment—a "Luciferase"—but as being essentially bound up with the vital processes of the cells, and dependent for its production on the intact organisation of the cell. These attempts to study by frigorific methods the physiology of the cell have already yielded valuable and encouraging results, and it is to be hoped that this line of investigation will continue to be vigorously prosecuted at the Jenner Institute.

And now, to conclude an Address which must have sorely taxed your patience, I may remind you that I commenced by referring to the plaint of Elizabethan science, that cold was not a natural available product. In the course of a long struggle with nature, man, by the application of intelligent and steady industry, has acquired a control over this agency which enables him to produce it at will, and with almost any degree of intensity, short of a limit defined by the very nature of things. But the success in working what appears, at first sight, to be a quarry of research that would soon suffer exhaustion, has only brought him to the threshold of new labyrinths, the entanglements of which frustrate, with a seemingly invulnerable complexity, the hopes of further progress. In a legitimate sense all genuine scientific workers feel that they are "the inheritors of unfulfilled renown." The battlefields of science are the centres of a perpetual warfare, in which there is no hope of final victory, although partial conquest is ever triumphantly encouraging the continuance of the disciplined and strenuous attack on the seemingly impregnable fortress of Nature. To serve in the scientific army, to have shown some initiative, and to be rewarded by the consciousness that in the eyes of his comrades he bears the accredited accolade of successful endeavour, is enough to satisfy the legitimate ambition of every earnest student of Nature. The real warranty that the march of progress in the future will be as glorious as in the past lies in the perpetual reinforcement of the scientific ranks by recruits animated by such a spirit, and proud to obtain such a reward.

ADDRESS TO THE CHEMICAL SECTION
OF THE
BRITISH ASSOCIATION.
BELFAST, 1902.

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President of the Section.

(Continued from p. 147).

THE contention that chemical equality must be regarded as of as clearly defined a nature as gravimetric equality becomes the more weighty when it is reflected that our very definite views concerning gravimetric equality are due solely to the law of conservation of mass, the evidence for and against which, I may remind you, is just now to be discussed by Lord Rayleigh before the Physical Section. The mass of one pound of sodium remains unchanged when the metal is converted into salt, washing soda, or borax; if this were not the case, gravimetric equality would be just as definite as it is now, but physicists would have to argue for its general recognition in much the same way as I am doing now for the recognition of chemical equality.

In further justification of this claim of chemical equality to co ordinate rank with dynamical equality in the quantification of substances it may be well to take the fact into consideration that the determination of the former is independent of that of the latter. Overlooking the difficulties of the task, let there be at hand or always procurable unlimited numbers of parcels of the different substances to be experimented upon, each of which, by other means than weighing, such as spatial measurement, can be known to be equal to, or greater or less than, other parcels of the same substances. Suppose, now, that after many trials, one of a number of equal portions of sodium hydroxide has been found to be the quantity just necessary to interact with one of a number of portions of hydrochloric acid also equal among themselves. The products of the interaction will be some water and some salt. We can now have placed before us a parcel of sodium hydroxide equal to that previously used, another of hydrochloric acid, also equal to that used, and the water and the salt obtained, and then have before us chemically equal quantities of four substances. Let now, by spatial measurement, a number of parcels of water be portioned out, all equal to that of the water obtained, and a number of parcels of salt equal to that of the salt obtained. By a series of trials we find a quantity of silver nitrate just sufficient to interact with sodium chloride, and having, by supposition, taken this quantity of silver nitrate from a lot of other parcels equal to it, we find that one of these is just sufficient to interact with one of the portions of hydrochloric acid equal to that used in producing one of the portions of salt. Further, we find that the salt and the hydrochloric acid each produce a substance which is the same, namely, silver chloride, and in the same quantity as the other. Along with it in the case of the salt, is sodium nitrate, and in the case of the hydrochloric acid, nitric acid. We can then find that this quantity of nitric acid is just enough to interact with one of those of sodium hydroxide, and thereby produce quantities of sodium nitrate and water, respectively equal to those obtained in the other interactions. If now we conjoin with these experiments others in which hydrogen, sodium, and silver are each caused to combine with chlorine, and others in which hydrochloric acid, silver chloride, and sodium chloride are electrolysed into these elementary substances, evidence is obtained of such facts of chemical composition and decomposition and of double decomposition (or what happens when compounds interact) as those upon which the science of chemistry is framed.

In teaching chemistry the point is kept too much in the background, if not altogether out of sight, that the chemical equality of quantities of different substances is

independent of all other relations of equality between them, and that, therefore, its validity is not affected by the fact of its terms agreeing with some and not with other terms of equalities determined in other ways. Instead of bringing out this point the molecule of water is given out as being, primarily and prominently, that quantity which has eighteen units of mass, and which measures two unit volumes. Both statements happen in the nature of things to be true, but neither of them describes the molecule. Let it be clearly understood from illustrative examples what is meant by "chemically equal," and there is hardly more to be said as to what constitutes a molecule of water than that it is the quantity of it chemically equal to that of some other substance presenting itself for comparison. "Molecule" is a term of relation: it stands for an equal quantity, not for any particular quantity; but as such it is as easy to understand and as indefinable as an equal volume or an equal weight of a substance.

It is then only as colligated equalities, established by experiment, that gaseous volumes, osmotic pressures, and other properties of substances come into consideration, first as enforcing the truth of the conception of the indicated quantities as equal, and then as the means of molecular measurement without resort to chemical change. But of the purposes served by the colligative properties, that of giving molecular measurements without recourse to the evidence afforded by chemical change is well known to be of the very widest application. To determine chemically the molecular equalities of substances, single chemical changes of suitable character, changes which are cases of double decomposition, have to be looked for; and to know these with the desirable degree of certainty calls for a much larger acquaintance with the chemical behaviour of the substances than can usually be gained at the early stage of work when the knowledge of the molecule is of the utmost assistance in the further investigation of the nature of the substances. Consequently, it is nearly always through recourse to physical methods that the molecule is first ascertained, and then through the molecule the certainty acquired that some particular interaction is a single one, thus reversing the normal order of things, which undoubtedly is that the molecule in chemistry, however it may have been first determined, is recognised as such by being what it is in chemical change.

I shall have been wholly misunderstood by you if you suppose that I would make light of the importance of the balance in chemical operations, or of the value of its indications in chemical investigations. Once the weights of molecular or atomic quantities have been ascertained, the balance becomes the most accurate and generally the most easily applied instrument for apportioning substances in these quantities. Chemical interaction, to be employed in this way and without the aid of the balance, is practically useless, for the reason that it involves the destruction of the quantities it measures. Out of this dependence on the balance arises the exceeding importance of accurate tables of atomic weights, from which molecular weights are derived by addition; but the place for these tables is not on the walls of the lecture-theatre, but in the laboratory pocket-book, and, perhaps, in the balance-room. Besides the use of the balance and of atomic weight tables for getting and calculating out molecules of different substances at pleasure, there is the indispensable service they perform in enabling chemical analysis to be carried out and applied to the solution of the problems offered by chemical change. The primary problem of every science is to find some element of sameness in the diversity of its phenomena, in order that they may be compared, a problem which was solved for chemistry to a large extent by Dalton, and ceased to exist when the distinction had been made between molecule and atom. But this having been solved, there comes the other problem, namely, to find definite, that is, quantitative differences in the midst of the uniformities,

and these for the chemist are differences of mass or weight. Through that redistribution of mass which attends chemical interactions it has been possible to trace out to some extent the nature of the transformation of substances and develop the science on the lines of chemical composition and chemical constitution. Thus, then, the balance has become and will continue to be the necessary instrument of chemical research; but again I would remind you that it records its facts in units which are not ours, and of which we avail ourselves only as the means to an end. Sodium chloride is chemically composed, not of 3545 equal parts of chlorine with 2305 of the same equal parts of sodium, but of equal quantities of these simple substances.

The theory of chemical molecules or equalities and their relations to the equalities between the weights and gaseous volumes of different substances were brought to light not by Richter's law of chemical combining proportions, and not by Avogadro's hypothesis as to their being equal numbers of particles in the same volume of different gases, but in the first place by Dalton's atomic theory and Gay-Lussac's law of simply related gaseous volumes in chemical change; and then, much more fully in the middle of the last century, through the brilliant work of Gerhardt, Williamson, Laurent, Odling, Wurtz, and others, in the purely chemical field. Dalton gave us the conception of the molecule, though confused with that of the atom, as the unit of measure of chemical activity in place of the gravimetric unit; the work of the chemists of the last mid-century gave us a fuller conception of the molecule, along with the notion of chemical change as being substitution in the molecule effected by what became known as double decomposition. Up to that time chemistry had been treated only as the science of compounding and decomposing or reducing. Sodium added to oxygen gives soda, sulphur added to oxygen gives sulphuric anhydride, soda added to the anhydride gives sodium sulphate, ethylene added to chlorine gives dichlorethane, water subtracted from alcohol leaves ether, and so forth. All this is strictly true in a limited way, but then it is not chemistry; and the addition precedes and does not constitute the chemical union. In the sodium sulphate we perceive no soda, no anhydride, no sodium, sulphur, or oxygen. That is to say, there is evidence of the addition and subtraction of mass and some other such evidence; but, for the rest, evidence of addition there is none. Were it otherwise there could be no chemistry. It is true that one of the great things accomplished by chemistry has been that of establishing the law of the conservation of mass, without which to rely upon the chemist would be unable to carry on his experimental investigations. But that is only because, like the steady point to the seismologist, it is there unchangeable when all else is changing. Since it is the law of no change, it cannot serve to explain what is change. Far from being the science of the composition of substances, chemistry might be defined as being the science of the non-composition of substances where that composition might have been looked for from the antecedents. If salt is verily a compound of sodium and chlorine, and can be broken up into these, why have the fragments not the marks on them of that whole of which they formed a part? It is true that 5850 parts of salt become 3545 parts of chlorine and 2305 parts of sodium, nothing being gained or lost in weight; but to account for that there is no need of chemistry, a science which takes cognisance of the phenomena of change, and not of those of unchanged properties. The use of the word "composition" in chemistry cannot be discarded now, and all that is necessary to make it unobjectionable is to see that the term is always qualified by the prefix "chemical" when there is a possibility of mistake about its significance, and that that significance is carefully explained, if not defined and fully illustrated, before it is given over to the beginner.

The facts of a chemical nature about common salt which

cause the statement to be made that it is a chemical compound of chlorine and sodium are such as these. Salt can be wholly changed into sodium and chlorine; these substances brought together change into salt and nothing else; salt and sodium, each under conditions appropriate to it, change into the same substance, called also a sodium compound, such as sodium hydroxide; salt and chlorine, each in its own way, change into the same chlorine compound, such as hydrochloric acid; neither sodium nor chlorine, one apart from the other or the other's chemical compounds, ever changes into salt; salt is, directly or indirectly, producible in the chemical interaction of a sodium compound with a chlorine compound; the properties of salt are much less like those of either sodium or chlorine than like those of some other substances; in sensible and other physical properties the chemically compound substance, salt, is as simple as or simpler than either of the chemically simple substances, sodium and chlorine; lastly, the laws of combining proportion by weight are obeyed in all the chemical changes in which salt takes part.

With exclusive reference to such facts as these, the chemical composition of a substance will, I think, be found to be satisfactorily defined, as its having the power, capacity, or property of being wholly producible from and wholly convertible into, directly or indirectly, those substances of which it is said to be composed. A simple substance differs from one that is compound only in not possessing the power of being by itself convertible into two others, or of being produced alone from any two others. Simple substances are not less varied or less complex in their physical properties than compound substances, while their chemical constitution is often more problematic than that of many which are compound. The term "simple," therefore, is as misleading in the language of chemistry as "compound," unless defined and qualified in use by the word "chemically."

The ground really occupied by chemical composition in theoretical chemistry is now greatly limited; for with the full acceptance of the idea of the molecule and of the atom as a derivative of it, its place has been taken by chemical constitution to an extent hardly realised. The useful and practically necessary expression of the results of the quantitative analysis of a new substance gravimetrically is all that can strictly receive the name of its chemical composition. When the term is applied more widely it is used for what are really the simpler forms of chemical constitution. It was otherwise before the conception of the molecule had become current and the atom had become a derived function of the molecule. Chemical composition as expressed by Dalton in atoms is indeed that and nothing else. Carbonic anhydride is composed, according to him, of two atoms of oxygen to one of carbon, as against carbonic oxide which is composed of one; marsh gas of two atoms of hydrogen to one of carbon, as against olefiant gas composed of one. But then it was only numerical necessity which led him to adopt such a mode of expressing the facts. The same necessity, it is true, affects us also in the matter of carbon dioxide, of water, and of ammonia, but how little it does so is shown by the many cases in which the empirical or simple composition is expressed in multiples. The atomic chemical composition of ethylene is two of hydrogen to one of carbon, and that of benzene one of hydrogen to one of carbon. When we say, as we always do, that the one substance is "composed" of four atoms of hydrogen to two of carbon, and the other of six of hydrogen to six of carbon, we give what is information concerning the constitution of these substances. Call it the composition of the molecule as we may, it is evident that by composition we can here mean only constitution. As with polymerism, so with isomerism, and in a more marked way. Mercurous sulphate and mercuric oxy-sulphate, quite distinct salts, have yet the same composition.

In the great reformation wrought by the chemists to

whom I have referred, but by Gerhardt in particular, the new light set up in chemistry was the notion of what came to be called "double decomposition" in chemical change. The phrase is not, perhaps, happily constructed, but it has the merit of needing some explanation of its meaning before it can be understood, and troubles, therefore, through a too simple apprehension of the sense of the word "composition" are hardly to be feared. Its introduction into chemistry marked the ascendancy of the idea of the molecule as the factor in chemical change whose interactions with other molecules were to be considered, instead of those additions which, as chemical phenomena, never take place. It led also to new conceptions of the nature of the atom and the compound radical as being the quantitative and qualitative expressions of the powers possessed by substances to change into others, and to the conception of the valency of atoms and radicals as expressing the nature of the connection of successive chemical changes. The zeal with which it was attempted to force all chemical changes into the form of double decomposition interfered, perhaps, with the full recognition of its importance; but the fact remains that, with hardly an exception, all that is stated concerning the nature of those chemical changes in which two or three substances become one, or one becomes two or more, is based upon notions derived from the study of double decomposition.

The fundamental value of double decomposition consists in its displaying threads running through chemical transformations which can be followed up. When two substances change into two others, and only then, there can be found, in most cases relations of resemblance, both physical and chemical, between the before and after of a chemical change. Instead of the striking unlikenesses shown by the substances formed by quasi-addition to those from which they are formed, there are here met with the similarities of the outcoming to the interacting substances, and the similarities between the products of different interactions in which the acting substances are similar. Chemists had been for very long familiar with acids, bases, salts, without becoming deeply impressed with the significance of the resemblances which these class-names imply, and also with the facts that acids beget acids, bases bases, and salts salts, or in more general terms, that substances in interaction produce others like them, and that differences between the products and the agents in one change are distinctly repeated in a similar change in which other substances are concerned, points now given expression to by such terms as "chemical constitution," "homologous," and "analogous series," "Kopp's law," &c.

What is so important to consider in the study of double decomposition is that the fact, that the sum of the masses of the two products of the change is the sum of the masses of the two interacting substances, presents itself no longer as being merely the evidence of the massing together of substances into a compound; for there is in double decomposition to be considered that redistribution of mass which, on the one hand, is found to correspond to, and be part of, a general though not sharply defined re-distribution of physical and chemical properties; and, on the other hand, to be obviously irreducible to that interchange of those simpler substances which in many cases are produced in the simple decomposition of the acting substances.

The physical properties of substances, or rather their sensible qualities, are of too uncertain a character for their redistribution to be safely traced. But it generally does result, amongst inorganic substances, at least, that colour is transmitted, the saline, acid, bitter, or other taste of one of the active substances will appear, with more or less distinctness, in one of the products, a relatively volatile and a relatively fixed substance together will yield a similar pair of products, a dense and a light substance will yield a dense and a light substance, and so on. The chemical properties, however, are quite definitely re-

distributed to a large extent, a fact sufficiently illustrated by saying that an iron salt yields an iron salt, and a sulphate yields a sulphate.

But this is not a redistribution in which simpler substances, or indeed any other substances than those interacting, play a part; as soon becomes evident on attempting to establish the contrary by an appeal to the facts. While silver acetate and silver sulphate resemble each other and also silver nitrate as silver salts, they do not resemble silver itself; and though silver nitrate resembles sodium nitrate as nitrate, there is not even a substance known which is related to these salts as silver is related to silver salts. It might be objected to this that there may yet become known such a substance, which in its ultimate decomposition would give one molecule of nitrogen to three molecules of oxygen. If instead of nitrate were given acetate or cyanide, there would be found in the substances acetic peroxide and cyanogen, it might be said, the analogues of the as yet unknown substance of the nitrate. But the point I would make is, that nitrate, sulphate, &c., are names with well-defined meanings independent of the fact that the corresponding substances are not known; for it follows without argument that also the terms silver, iron, chloride, &c., should be equally independent in meaning of the existence of the substances silver, sodium, chlorine, &c. It is a familiar historical fact that cesium, helium, and fluorine were chemical names long before the substances cesium, helium, and fluorine became known. We might well be convinced, therefore, without going further, that constitutional names, names which convey the facts of likenesses preserved in chemical change, cannot be indicators of the presence of the substances for which they may be also used. For, that being the case, we have no grounds for assuming that silver nitrate in interaction with sodium sulphate decomposes into the substance silver, which then combines to form silver sulphate. But fuller proof than any appearance of likeness or unlikeness can give is afforded by facts which became known and appreciated in connection with the chemical molecule. Typical of them all is the fact that in none of its interactions does chlormethane yield a hydrocarbon simpler than methane or than itself. Under those conditions in which it might have been expected to give a substance which would be methyl, it produces ethane, a substance which chlorine converts into another substance, having instead of one-third only one-sixth less hydrogen in its composition. Similar results have been obtained in all cases where the point can be determined—that is, where the simpler substance looked for would still be a compound substance, and such simpler derivatives are looked for no longer. The monohydride of oxygen or sulphur, the dihydride of nitrogen or phosphorus or arsenic, the mononitride of carbon, the organic compounds, methyl, phenyl, acetyl, are not only unknown, but are held to be non-existent substances, though their chemical compounds, the hydroxides, amides, cyanides, and the rest, are both numerous and well-defined. Whatever other view we shall have to take of the constitution of Gomberg's remarkable "triphenylmethyl," it will certainly not be that it is identical with the radical of the triphenylchlormethane from which it is derived, unless we are prepared to allow that carbon is sometimes tervalent. Ethylene the substance differs from ethylene the radical in having its two carbons differently related; but it is difficult to see how to make a similar distinction in the case of Gomberg's substance.

In those other cases in which the point is not strictly determinable, only because the resulting substances are the simple substances themselves, it required but the recognition of molecular quantities to make it evident that these cases run parallel with the others. For, in all changes which can be satisfactorily followed out, the resulting or entering quantity of the simple substance is twice as great as that which can have come from, or gone to form the molecule of either of the compound sub-

stances. But if, so far as can be traced, a simple substance comes only half from one molecule of any of its compounds, none of these compounds can contain or be composed of simple substances. All simple substances, therefore, as well as all compound substances, enter into, and come out from chemical changes as dual in all of them in origin and disappearance. Their colligative properties have been appealed to in order to confirm this observation, but with conflicting results, sometimes confirmatory of the chemical evidence, sometimes contradictory of it, and sometimes too complex for confident chemical interpretation.

I refer here more especially to Avogadro's proposition, which is in effect that equal volumes of gases are chemically equal or molecular. As in the case of Dalton's atomic theory, there is to be distinguished in this proposition what Avogadro really put forward as new from what he took for granted. Admitting, as was to him a matter of course, that gases have in equal volumes equal numbers of particles, he asserted that in the case of elementary substances these particles, are not the atomic particles, but, as in the case of compound substances, particles compounded of these, which interact with the particles of other gases as chemically equal each to each. If now this proposition is divested of all hypothesis, all reference to the mechanical structure of gases, it becomes the law that equal volumes of gases at the same temperature and pressure, whether simple or compound, are almost exactly chemically equal quantities, and once in possession of this law we find nothing becomes clearer by assuming that equal volumes of different gases contain the same number of chemically equal particles. This law is, obviously, an advance upon Gay-Lussac's law similar to that of the chemical molecular theory upon the atomic theory of Dalton. Unfortunately, however, it does not hold good in the case of not a few simple substances, and it seems impossible from the chemical point of view, and consistently with the molecular theory, to admit that, because the gas-volume has only half the expected mass, the chemical molecule of sodium or mercury is not bipartite like that of hydrogen or oxygen, and chemically equal to either.

The dual constitution or chemically compound nature of the simple substances as thus established by the part they take in chemical interactions furnishes further evidence of the untenability of the belief that the molecule is chemically composed of two substances, or their substitutes, simpler than itself, when we consider that, were this true, there would be chemical union between two things perfectly alike, two portions of the same thing. This difficulty was, I believe, first raised by Berzelius, and has never been met. Physically, the matter is simple enough, if motion in the opposite direction is not counted as a difference between two masses. But this would be a non-elective union, whilst chemical union is elective.

The difficulty, insurmountable when made, does not arise when the fact is recognised that every chemically single substance, whether simple or compound, is, as a substance, one and without parts, and can never, therefore, be built up of or broken down into parts different from itself. One substance (as two molecules) or two substances change into two others or into two molecules of one, in an interaction which is instant, uninterrupted, and irresolvable into stages, where the interaction is single in character. But just as a body can be mentally analysed (as in the investigations of dynamics) into mass and motion, which apart are unknown, and as these again can each be conceived of as further divided, resolved, condensed, and otherwise qualified as centres of mass, compounded motions, and so forth, so the chemist is enabled mentally to find quantitatively defined this, that, and the other mark of the many chemical interactions which have or may have gone to bring it into existence, and will or may again have place in the possible forms of its dissolution into others. The two methyls in the constitution of ethane, about which we are quite certain, are not

two things held together till some interaction sunders them in the chemical dissolution of ethane, but the double mark of similarity between it and other methyl compounds in their chemical interactions. We cannot say that only one part of the ethane is methyl, or hydrogen, or carbon, but that part of its nature, of its constitution, is its behaviour as a methyl compound, or, again, as an ethyl compound; or, more comprehensively but less specifically, part of its constitution is its behaviour as a hydrocarbon, as a hydrogen, and as a carbon compound. But these are different aspects of it, different relations of it, not differing parts of the one homogeneous substance.

(To be continued.)

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING AUGUST 31ST, 1902.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolitan Water Act, 1871.

London, September 10th, 1902.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 192 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from August 1st to August 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 192 samples examined by us chemically during the month, all were found to be clear, bright, and well filtered.

The rainfall at Oxford still shows a deficit, but has been fairly well distributed throughout the month, rain having fallen on twenty days. The total fall was 2.20 inches, and the average is 2.38 inches; this leaves a deficit of 0.18 inch, which, added to the previous one of 5.17 inches, makes a total deficit of 5.35 inches, or 34.1 per cent on the thirty-five years' average.

Our bacteriological examinations of 499 samples have given the results recorded in the following table; we have also examined 36 other samples, from special standpipes, wells, &c., making a total of 535 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 24 samples) ..	304
New River, filtered (mean of 126 samples) ..	13
Thames, unfiltered (mean of 24 samples) ..	5134
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 269 samples) ..	21
Ditto ditto highest	193
Ditto ditto lowest	0
River Lea, unfiltered (mean of 24 samples) ..	233
River Lea, from the East London Company's clear-water wells (mean of 32 samples) ..	33

Of the 427 samples taken daily from the filter-wells of the Metropolitan Water Companies, and examined bacteriologically by us, eighteen samples, or 4.2 per cent, were sterile. Three samples contained more than 150 mi-

crobes, while only twelve samples, or 2.8 percent, contained more than 100 microbes per c.c. The mean number of microbes in the twelve excess samples was 142, against a corresponding mean of 149 in twelve samples during July. These figures show that the Metropolitan waters are keeping well up to their usual high standard of the summer supply.

We are, Sir,
Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

CORKS VERSUS RUBBER.

By T. H. PAGE, B.Sc.

As it is well known to those who work with essential oils, the terpenes, especially when hot, very rapidly attack rubber corks, making them first swell and then become very soft and sticky. This is most objectionable, not only because they are disagreeable to handle, but they leave rubber on the necks of the flasks, &c., and it will often get into the distillation flask and even into the condenser tube, discolouring and spoiling the distillate.

Having had occasion to carry out a lengthy fractionation of terpenes under reduced pressure with a Young's dephlegmator, I found it practically impossible to use rubber stoppers, as they were not only in contact with the hottest vapours, but sometimes even with the liquid itself, and would have to be frequently renewed.

To overcome these difficulties I find an ordinary cork covered with gum an excellent substitute. The gum used is the ordinary gum mucilage, but rather thick. It is conveniently applied with a brush. The apparatus is fitted together, exhausted, and the cork brushed over with the gum several times till a satisfactory joint is obtained. In this way as good a vacuum is possible as with rubber. Working with an ordinary Bunsen pump there is no difficulty in getting 12 to 20 m.m. pressure, according to the water temperature.

Big holes, which sometimes occur, especially in large corks, are best stopped with shellac or gum slightly softened in water.

The gum is not affected by the terpenes, but hardening as the distillation proceeds, forms a perfectly air-tight covering to the cork.

In cases where the joint is frequently broken, as in repeated re-fractionation, the gum remains somewhat soft and flexible, and by simply brushing it afresh the joint is very rapidly made.

It is, of course, with the large corks that this method is of the most use and value. I have used it with corks up to 1½ inches diameter.

The same plan answers equally well for where the delivery-tube of the flask goes into the condenser, and for the cork holding the thermometer. If the neck of the distilling flask is perfectly straight and not turned over, the corks have a tendency to slip in, but this is readily avoided by cutting down a cork, rather too large, so as to form a ridge which rests on the edge of the neck.

For workers in essential oils this is invaluable; the obvious advantages being:—

1. The corks and gum are not affected by the oils, nor are the oils affected by them.
2. The corks are not spoilt, and can be used repeatedly. I have had one in use for over three months.
3. The joint is quickly made and is perfect.
4. Last, but not least, the difference in cost is very considerable, especially with large sizes.

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J. A. Wood.—Any good makers of chemical apparatus will supply you with the distilling plant you require. A book has been written by Mr. F. Nutt on "Receipts for Home-made Wines, Cordials, &c." We do not know the publisher's name.

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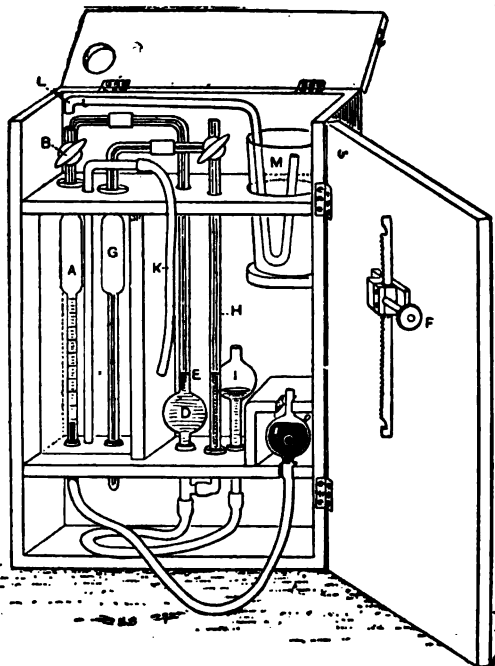
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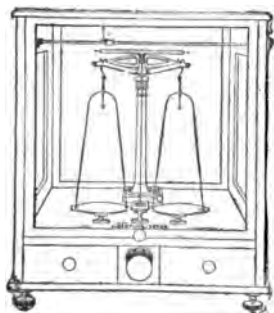
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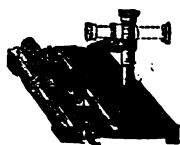
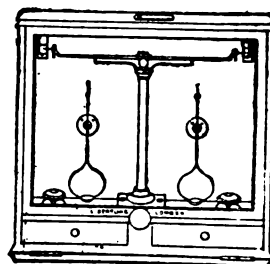


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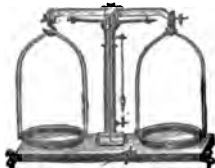
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ADDRESS TO THE CHEMICAL SECTION OF THE BRITISH ASSOCIATION.

BELFAST, 1902.

By EDWARD DIVERS, M.D., D.Sc., F.R.S., V.P.C.S.,
Emeritus Professor of Chemistry in the Imperial University of Tokyo,
Japan.

President of the Section.

(Concluded from p. 161).

WITH the laudable object of combating the prevalent notion that matter is something which is the basis or essence of a body, something acting as the medium of the manifestation of its forms of energy, a distinguished and most lucid writer on chemistry has, adequately perhaps for that object, represented a body as a compound of the various forms of energy subsisting together and cohering in certain proportions within the volume of the body. But this presentation of a subject as a cohesion or association of forms of energy is on the same footing as the presentation of ethane as consisting of two methyls bonded together, or two portions of carbon with six of hydrogen. It is compounding what cannot be had apart, what cannot be even conceived of as separate, so far as bodies are concerned. The analysis of bodies into manifestations of different properties are only mental operations. A moving body, a hot body, a green body, an explosive body, becomes by legitimate abstraction a phenomenon of motion, of heat, of colour, or of light, or a chemical phenomenon as our needs require; but the body is there all the while, and its undivided and continuous existence is indispensable to the phenomenon. The body can be hotter or colder, but not that only,—not that without other differences; red-hot iron is throughout a very different thing from cold iron, and ice differs widely from steam in most of its properties. A substance is no more composed of its properties or energies than it is composed of its so-called elements. It manifests its presence in a thousand and one ways more or less distinguishable; its properties are so to manifest itself. But no divisibility of itself while it remains itself can be thought of, no differentiation can be suggested, no nucleus with its superinduced properties can be traced.

It ought, therefore, to be possible to express all the particulars of chemical constitution without making any assumption as to substances having parts or structure. Of chemical constitution itself, I doubt whether there is to be found a definition which is not couched in language having reference to the minute mechanical structure of substances, notwithstanding the fact that all knowledge of their chemical constitution has come to us through observation of the properties of the substances themselves, and more particularly their relations in cases of double decomposition. Bearing in mind that all terms are relative, I think the chemical constitution of a substance may be defined as the resemblances shown by it in its chemical changes to other substances, often better known than it and taken as types, these resemblances being indicated and described usually by means of special nomenclature and notation. As this nomenclature and notation have been developed out of those designed to express chemical composition, it is well to point out that the notion of chemical constitution is independent of that of the latter, though clothed to some extent in its language and symbols.

The notions of radical and atom are so intimately related as to be often used indifferently, the one for the

other. The radical, ethylene, is always an atom of ethylene, the radical nitrogen always an atom of nitrogen. Radical and atom are in fact the qualitative and quantitative aspects of the same thing. They are thus exactly parallel with substance and molecule. We can think of unquantified substance, and perhaps of unquantified radical, but in chemistry we never really want such conceptions; one of the many definitions of science is the quantification of phenomena, and in every chemical phenomenon the substances concerned are quantified as molecules. The quantification of radicals expressed by the atom is fundamentally the same in principle as that of substances, namely, that of chemical equality in interaction; but it may be better to say that it is dependent upon the quantification of substances as molecules.

In the interaction of double decomposition each substance by contact and union with the other develops and manifests a dual character by becoming distributed as the two new substances, with the consequence that each of these has certain properties the same as those of the one, and certain others the same as those of the second interacting substance. What is common in this way to one of the interacting and one of the resulting substances is a radical of these substances, of which there are evidently four in every double decomposition. These radicals of a single interaction are defined as whatever two parts of the powers of a substance to yield the simple substances of its chemical composition are, in certain interactions, continued separately from each other in the two new substances. But the pair of radicals developed in the various double decompositions of a substance being by no means always the same, one of the radicals of one pair must include in its composition part or all of one of those of another pair. Acetic acid has for one pair of radicals methyl and carboxyl, and for another pair acetyl and hydroxyl. Of these, carboxyl includes hydroxyl, and acetyl includes methyl. Again, acetic acid yields the hydrogen and acetate radicals in one interaction, and hydroxyl and acetyl in another, so that in these cases the acetate radical includes acetyl and the hydroxyl includes the radical hydrogen. Now, what is common to carboxyl and acetyl and what is common to the acetate radical and hydroxyl are also treated as radicals, the one being known as carbonyl and the other as the radical oxygen. These are examples of what may be distinguished from the others as the polyvalent radicals. They are radicals of radicals, and therefore also radicals of substances. They may be defined as the common part of two or more other radicals. A single definition of all radicals can be given, but it is not instructive. A radical is any single power or any interdependent association of the powers of a substance to produce simple substances which continue in any product or series of successive products of its chemical change.

Before I leave the subject of the radical I wish to repeat that it is only when it is interacting that a substance shows a dual character or division, as it were, into parts or radicals, and that the duality it then shows is determined as much by the nature of the other substance as by its own. A substance is neither actually or conceptually the sum of its radicals. The very fact of the difference of these in different interactions should be proof of this; though it only leads to its being taken to be at least the sum of its ultimate or simple radicals. If, however, it is not the sum of its proximate radicals, it is hard to see how it can be imagined to be that of the ultimate ones. In relation to its radicals, a substance must be held to present itself as any one of these for the purpose of investigation, and at the standpoint from which it is considered. It is then to the mind that particular radical, though also something else; just as snow is white and cold, yet also something else, for the moment unconsidered. Nor can the two products of an interaction be looked upon as themselves the sum in properties of the interacting substances. To a limited extent and imperfectly, we can attach to a given radical certain of the properties common

to its compounds; but it needs no greater insight than we have already, to recognise that a substance cannot be what it is in one way, without being in that way greatly affected by what it is in another. This is now a recognised but not sufficiently considered point, and I therefore welcome those publications of Professor Vorlaender, of Halle (who now honours this Section with his presence), in which he has been vigorously calling attention to the extent to which the properties of a substance, acid, basic, stable, and what not, depend as much as, if not more, upon the interrelations of the radicals than upon the radicals themselves.

One other thing I have to say about the radical, which is as to the spelling of the word. I plead for a return to the ending of the word radical with "al," now interdicted in the *Journal of the Chemical Society*. It seems appropriate to call the powers of a substance to behave chemically as it does, the roots or radical parts of its chemical nature, but it does not seem appropriate to call them radicles or rootlets. Americans and all other nationalities but our own use the original spelling.

I have put off too long, perhaps, all reference to the properties of very dilute aqueous solutions of salts, but I wished first to discuss the nature of the radical. The osmotic pressure and other dependent points which are particular in the behaviour of such solutions are in full accordance with the assumption that an electrolyte by dissolution in much water becomes a pair or a binary system of two interdiffused quasi-substances called "ions." These ions must differ from isolated substances in bearing equal and opposite quantities of electricity; in being each unknown apart from its fellow; and in having a composition not to be found in actual substances, though identical possibly with that which a radical would have were it a substance. The ions can be indeed separated from each other, but not to continue as themselves, since in the act of separating they form ordinary substances, either by uniting with other ions, or by two molecules of ion becoming one molecule of substance. In the former way of separation the ions of two salts interact on mixing their solutions; in the other way, the ions become substances when their solution is placed in a galvanic circuit. In this mode of separation—by electrolysis, that is—the substances corresponding with the two ions, or else secondary products of their change, are produced, the one substance at the kathode and the other at the anode, while the solution away from the electrodes, but between them, remains for the time unaltered in composition. Along with this there occurs in many cases a phenomenon first recorded by Daniell, and afterwards investigated by Hittorf with such beautiful results. This consists in a greater fall taking place in the concentration of the salt solution close to one electrode than in the concentration of that close to the other, as though the ions were hydrate compounds, and that the one ion was a higher hydrate than the other. Until we know more of the nature of the ions themselves this phenomenon is most conveniently quantified on the hypothesis that the ions travel as molecular particles, but the discussion of this hypothesis is beside by present purpose.

The phenomena of ionisation or, in other words, the particular properties of dilute solutions of salts, belong evidently to a change unlike all other chemical changes. It is a polarised chemical change, in which the equivalent and complementary products of the interaction appear apart and at remote surfaces of the mass of decomposing salt solution. Two points which call for notice in connection with my present subject are that an ion is one of a pair of quantities commensurate with the quantity of the salt itself that is or would be in interaction; and that it is molecular in character and therefore to be regarded as a relative and wholly variable quantity.

Dalton's atoms were both the atoms and the molecules of present-day chemistry, but much more the latter than the former. Although the chemical atom can now be no more than a dependency of the molecule, it is commonly

set up as the starting-point in chemical theory, and as having an independent existence as a quantity of the substance, while the molecule is represented as being a conjugation of atoms. But there cannot be two standards in reference to the same thing, and in molecular chemistry the atom must give way. As I have already had occasion to point out, the atom is of the radical, the molecule is of the substance.

The four radicals of a double decomposition are equal and chemically complementary. These chemically equal quantities of such radicals are atoms. The quantities of all other radicals are also atoms, but only those of proximate radicals, those of a single interaction, are equal. Similarly, the quantities of the four substances of a single interaction are all equal and are molecules, but the quantities of substances are not equal in other interactions. These others are treated as the simultaneous occurrence of two or more single interactions, which they can always be represented and sometimes demonstrated to be. Calcium hydroxide and hydrogen sulphide give calcium hydrosulphide and water by two single interactions together, which in this case can be easily distinguished, since the calcium hydroxide will also interact with only half as much hydrogen sulphide to form the insoluble crystalline calcium hydroxyhydrosulphide and half as much water as before; this calcium salt will then interact with as much more hydrogen sulphide as went to form it, and produce the very soluble crystalline calcium hydrosulphide. Or the calcium hydrosulphide and as much calcium hydroxide as yielded it will readily interact to form twice as much as the first-obtained quantity of calcium hydroxyhydrosulphide. Thirdly, the calcium hydrosulphide and half as much water as was formed with it from calcium hydroxide readily interact to produce calcium hydroxyhydrosulphide, and half as much hydrogen sulphide as was needed to form the hydrosulphide. Therefore, and on other grounds, we say and know that one molecule of calcium hydroxide and two molecules of hydrogen sulphide give one molecule of calcium hydrosulphide and two molecules of water. This is, of course, only the law of multiple proportions introduced into chemical interactions. The expression "two or more molecules of a substance" has a meaning only as indicating the number of simultaneous or successive single interactions which have led to the conversion of certain substances into others.

Now a similar but complementary state of things meets us in the case of radicals. Instead of the coefficients of molecules, necessitated by having to consider many chemical changes as being cases of two or more single interactions occurring together, there are the valency coefficients of the polyvalent radicals, called out also by such a compound interaction. Thus, in the above case, whilst the single interaction between hydrogen sulphide and calcium hydroxide shows calciumhydroxyl as one of the radicals, the succeeding interaction between the calcium hydroxyhydrosulphide and more hydrogen sulphide shows the radical calciumhydrosulphuryl, and the common part of these two radicals is the bivalent radical, calcium. It will be evident that to give the atom of the calcium radical as bivalent is a statement reciprocal or complementary to that of giving two molecules of hydrogen sulphide as interacting with one of calcium hydroxide. Chemical equality remains still the measure of the atom, but that, in complex changes, whereas the number of molecules of one substance marks the number of single interactions, the valency number of the atom marks the same thing for the radical. It is a matter of valency, and not otherwise a matter of the atom. The radical calcium is never actively bivalent in a single interaction; in other words, it is never equal to two atoms of hydrogen. As a simple radical it does not take part in such an interaction; but it does do so as a radical of radicals, such as calciumhydroxyl and calciumhydrosulphuryl, and then has the same measure as—is equal in exchange to—the atom of hydrogen, though carrying with it of necessity other

radicals, a thing the hydrogen radical never does or can do. To take another example: when acetamide is formed from acetic acid, the nitrogen of the amidogen and the oxygen of the hydroxyl are equal in exchange, but because of their valencies the one carries with it two atoms and the other one atom of hydrogen. This is no matter of merely academic contention, for upon its recognition rests the doctrine of valency itself.

The quantity of the radical is the only proper and sufficient definition of the atom, whether the radical be that of a single interaction, or a radical of radicals, that is, a polyvalent radical. The atom is, therefore, the quantified power of a substance, as the compound of the radical, to produce other compounds of the radical, including its compound with itself, where that is possible. As with the molecule of a substance, so with the atom of the radical, it is of no fixed magnitude, and may weigh a kilogram, just as well as only a milligram, or something much less. Being a relative quantity and nothing by itself, of its indivisibility there is nothing to be said outside its definition; whilst, as to its being the smallest relative quantity interchanging in an interaction, it had only thus to be defined when there was uncertainty as to the molecule and the single interaction.

It has been impossible for me to discuss the nature of the radical and the atom without referring to valency, but it is itself a subject of such importance as to need special consideration. It does not seem right to me to say even the little I can say about valency without naming with the respect they deserve from us the distinguished chemists who laid the foundations of the doctrine and developed it:—Williamson, Odling, Wurtz, Edward Frankland, and Kékulé. I had the good fortune to be in the same laboratory as, and then intimate with, Kékulé, when in 1854 he was working out the bivalency of sulphur and oxygen by his investigation of thioacetic acid, some time, that is, before he had thought out the benzene ring and the valency of carbon.

Only when, as is usual, propositions are made in which a separate and independent existence, with valency as a property, is imputed to a radical, does the question, as to what valency is, present any difficulty. Approaching it from the side of the molecule and of double decomposition, and therefore from the experimental side instead of from that of the radical itself, as is customary, valency presents itself as being the number of single interactions necessary, in order to have a certain radical occur, first as that of one substance, and then as that of another which has no other radical in common with the first substance. That ammonia possesses one atom of the radical nitrogen, and three atoms of the radical hydrogen, and that the nitrogen radical is trivalent and the hydrogen radical univalent are statements mutually based upon facts such as the following:—Potassium nitrilosulphate, which contains nitrogen but no hydrogen, is converted by water in a sharply defined single interaction, into potassium-hydrogen sulphate, and into potassium imidosulphate, a substance which contains all the nitrogen along now with hydrogen. This salt passes, also sharply and by a single interaction with water, into as much more sulphate along now with potassium amidosulphate, which latter substance contains all the nitrogen and twice as much hydrogen as belonged to the imidosulphate. Lastly, the amidosulphate interacting with water gives a third quantity of potassium sulphate, equal to the last, and also ammonia, having all the nitrogen of the nitrilosulphate started with, three times as much hydrogen as the imidosulphate, and nothing else. That is to say, the nitrilosulphate and the ammonia have no other radical than the nitrogen the same, while three single interactions have been necessary to separate in this way the nitrogen radical from the three atoms of the potassium-sulphonyl radical. Therefore the nitrogen radical is trivalent and its quantity is the atom. Again, there are three atoms of the univalent hydrogen radical in the ammonia molecule, because in each of the three interactions an equal quantity of this radical is brought in from water.

Ammonia shows only one pair of radicals, behaving, so far as its own interactions go, exclusively as a compound of amidogen and hydrogen, and these radicals are referred to as united or bound together in being ammonia. It is only the interactions of its derivatives, primary, secondary, and tertiary, that are indicated by treating the amidogen as ultimately nitrogen and two hydrogen radicals. But this involves the consideration of all three hydrogens as bound to the nitrogen; and it becomes, therefore, of vital importance to bear in mind that the hydrogen radical, proper to the ammonia itself, is bound to a nitrogen radical which carries also bound to it two other hydrogen radicals.

Chemical formulæ still remain to be considered. They are symbolisations of deductions from experimentally ascertained facts, and are independent of the interpretation commonly given to them as referring to the minute differentiated structure of substances. A chemical equation expresses a chemical change quantitatively by means of chemical formulæ which are molecular. In a case of double decomposition, therefore, there are four formulæ; but when two or more such interactions are expressed in one equation, because they occur together, the formulæ of transition-substances do not appear, and then numerals before formulæ tell the number of interactions in which separate molecules of the substance have taken part. A formula represents the relative interacting quantity or molecule of a substance, while the single symbols composing it stand each for an atom of the radical of a certain simple substance as possessed by the substance formulated. The connecting lines and dots, and certain collocations of the symbols, indicate the association of the simple radicals as compound radicals in different interactions.

What is symbolised by position formulæ, and indeed by the formula altogether, are the chemical activities and abilities of the substance and its derivatives, and their analogies with those of other substances. When not in interaction, a substance has no constitution and no formula. It is certainly not on any experimental grounds that it can be regarded as some spatial arrangement of unlike parts. To take the simplest case; if we start with sodium hydroxide and symbolise its molecule by some mark, such as X to begin with, the interaction of the substance with an acid leads us to replace the X by two symbols and a connecting mark. One of these will be Na for the sodium radical; let the second be Z for the other radical, and let a dot or stroke be placed between the symbols to mark them as those of a pair of radicals in interaction. In other interactions, such as that with melted potassium acetate, we find need for a new pair of symbols, one being H for the hydrogen radical, while the other may be Q. But it is easy to decompose two molecules of sodium hydroxide in one operation into molecules of sodium, hydrogen, and oxygen, from which fact we learn that Z is replaceable by the double symbol O—H, and Q by O—Na. Thus, Na—Z and H—Q become equally Na—O—H, which records the ultimate radicals of sodium hydroxide, together with all its interactions, immediate and remote. But it does this with no more implication of spatially placed and tied parts than is made by expressing the measured flow of time by a straight line, or than is to be found in t^2 seconds of time, or in c^3 as the third power of a number, unless we specifically condition this symbol as stereometric. A formula is not to be read—on experimental grounds, I mean—as a symbol of parts juxtaposed and joined on, and should be regarded as an intricate but legible monogram telling the chemical nature of the substance. Every symbol in it is to call to mind a phase of the chemical activity of the substance or of its derivatives, a phase that may be for the time as the substance itself to the investigator, just as a pigment substance becomes only a red or a white to the painter. For example, salt is often nothing more than its chlorine phase to the chemist when he wants only a soluble chloride; whether it is of potassium, sodium, or ammonium, then matters not to him.

The double linking of the carbons in ethylene is a symbolised expression of facts, without reference to hypothesis. The two carbon radicals of ethane or of alcohol behave together just as does the single carbon of methane or the nitrogen of ammonia in being, but with a valency of six, continued to other compounds devoid of all the other radicals of the ethane or alcohol—that is, of the hydroxyl and the hydrogens. The quadrivalency of each carbon is made up by the interaction necessary to dissociate or to bring together the two methyls, which counts as a unit of valency to each carbon. Ethyl hydrogen sulphate decomposes into sulphuric acid and ethylene, the hydrogen-sulphate radical with a hydrogen radical becomes the acid as the one product, while the methylene radicals again pair off as the two methyls had done when ethane was formed, thus producing the non-saturated substance, ethylene. Since there is a perchlorethylene, the second linking mark falls between the two carbons; and when ethylene passes back to an ethane compound two units of valency are displayed by it without the carbons becoming dissociated.

Position formulæ of isomerides, such as those of propyl alcohol and acetone, present no difficulty, because they are interpreted as the expressions of unlike double decompositions. It is not unfrequently the case that no constitutional or structural formula can be given to a substance which shall express all the pairs of radicals possible in its interactions, of which the best studied example is that of ethyl acetoacetate. This state of things, known as tautomerism, admits of no other interpretation than that there are really two substances existent, of which one only is known, the other or so-called "pseudoform" requiring the assumption of its existence as a transition substance only. The notion of the shifting hydrogen radical is but the hypothetical way of viewing the intervention of the intramolecular change by which the substance becomes its "pseudoform."

The cyclic formula of benzene expresses the fact that, unlike a fatty hydrocarbon, benzene shows but one pair of interaction radicals, hydrogen and phenyl. The "ortho-," "meta-," and "para-" positions in benzene derivatives are only expressions of facts of "position" isomerism, such as those pertaining to other non-saturated compounds, but more complex to unravel and more varied and interesting. It is doubtful whether the Kékulé ring does not remain as efficient a symbol as any stereographic substitute yet proposed for it; but it itself is purely a symbol of chemical interactions, and has no spatial significance other than what may be put into it by convention. "Adjacent," "opposite," and the like have only application literally to the arrangement of the symbols; but if the symbolisation is perfect the "opposite" carbons will, as a matter of course, always indicate the same point concerning the chemical interactions.

Whether the chemical formulæ for the lactic acids are better arranged in a plane or as a tetrahedron is to be decided by the facts concerning these and other asymmetric carbon compounds, the object being to symbolise or formulate as distinct and complementary in certain physical properties, but alike in their chemical interactions, two isomeric substances, simultaneously formed in molecular quantities. Enantiomorphous arrangements of the respective formulæ of dextro- and lævo-lactic acids fully meet the case, but the facts are in no way explained by these formulæ. In the enantiomorphously related hemihedral crystals of the corresponding salts of the dextro- and lævo-acids, and in their opposite rotatory effect in solution upon the plane of polarised light, we recognise something like a torsioned state of the whole homogeneous substance, something to be accounted for by peculiarity of chemical origin, but not something made more intelligible by any imagined arrangement of unlike parts. It is possible to give an account of the chemical facts without making reference to mechanical structure, and then to reason about them somewhat in the following way:—Given the case of a

substance doubly equipped with the power to take part in a certain interaction, and considering that the exercise of the power can only be single, and that it cannot be made without affecting and transforming, or perhaps nullifying, the second equipment with power, predict what will happen. This is the prediction called for concerning any interaction which generates an asymmetric carbon compound. The result could never have been predicted; yet how natural and beautiful it is when it comes to us through experiment enlightened by the genius of Pasteur, Le Bel, and van't Hoff! That answer is that a twinned substance results, one indeed in most respects and chemically, but two in certain physical properties, characterised by presenting phenomena as of equal and opposite strains, a polarised pair of substances, in fact. What I mean by the double equipment with power is, of course, the pair of identically related and self-identical radicals, or the bivalency of one radical wholly and directly associated with the carbon radical. The case of the oxygen radical of aldehyde is that of the bivalent radical; the other case is that of the two carboxyl radicals of hydroxy-tartronic acid, or that of the two methylene hydrogen radicals of alcohol which these carboxyls have replaced. The tetrahedral formula with its reflected form admirably symbolises the case of enantiomorphously related pairs of substances. But no light whatever is thrown upon the nature of this pairing by the tetrahedron model; its value depends upon the fact that as a symbol it so fully matches the constitution of the substances.

Here I bring my summary of chemical theory and its formulation without hypothesis to a conclusion, hoping that, to some extent, I may have impressed you with the fact that the exposition of even advanced chemistry, in its symbolic, equally as in its ordinary language and nomenclature, is independent of any hypothesis as to the mechanically and chemically differentiated structure of substances, and that chemistry can be studied and still further developed without reference to such a structure. I have asked for few or no reforms in the use of either terms or symbols, my point having been only to press for a consideration and discussion of the doctrines of chemistry and the great atomic theory itself as something concerned exclusively with experimental chemical facts.

THE SYNTHETICAL ACTION OF ENZYMES.*

By Dr. E. FRANKLAND ARMSTRONG.

It has long been known that enzymes have the power of inducing hydrolysis, particularly of disaccharides containing a "glucoside-linkage," but the observation of Croft Hill that enzymes can also effect synthetical changes is of quite recent date; his experiments have shown that when maltose is hydrolysed by maltase, just as in the case of the hydrolysis of esters by acids, a reversible action takes place in concentrated solutions; and that in concentrated solutions of glucose a disaccharide is formed. Some uncertainty exists as to the nature of this sugar, Hill claiming that it is maltose, whereas Emmerling contends that he has identified it by means of the phenylosazone with isomaltose. Bearing in mind the great difficulty experienced in purifying and characterising the osazones of disaccharides, it is impossible, on the strength of this one derivative alone, to decide the question; but as Emmerling has shown that the sugar formed is not fermented by "top yeast," it may be concluded that it cannot be maltose.

Croft Hill's conclusion that enzymes exercise synthetic functions has been justified by the later observations of Kastle and Loevenhart and of Hanriot on lipase, the

* Read before the British Association (Section E), Belfast Meeting, 1902.

enzyme by which fats are hydrolysed, these observers having shown that this enzyme is capable of producing both esters of fatty acids and glycerides. In these cases stereochemical isomerism is out of the question, and the products are identical with the natural substances. Emmerling has also demonstrated that the synthesis of amygdalin from mandelio-nitrile glucoside and glucose may be effected by means of maltase.

It is interesting to note that Fischer has proved that isomaltose, not maltose, is formed by the action of acids on glucose, showing that in their case the action is not strictly analogous to the reversible hydrolysis of esters by acids, and it is, therefore, the more probable that isomaltose should be formed by the action of enzymes, as their behaviour in other respects resembles that of acids. Unfortunately nothing is known as to the relationship of isomaltose to maltose, and it is impossible to say whether they are merely the α - and β -glucoside forms, or whether they differ as regards the carbon atom to which the glucoside linkage is attached.

The author has been engaged in studying lactase in conjunction with Professor E. Fisher, and they have been able to demonstrate that this enzyme is one of those which are capable of inducing synthetical changes.

It was found that, whereas the action of lactase on milk-sugar was at first hindered and ultimately stopped entirely by the presence of the monosaccharides to which it gives rise; when the amount of these was further increased a change in the reverse direction took place. Accordingly the enzyme was kept for some time at 35° in a concentrated solution containing equal parts of galactose and glucose, toluene being added as an antiseptic. Both the reducing power and the optical rotatory power of the solution diminished steadily, and the phenylhydrazine test showed that a sugar was present capable of forming an osazone which was easily soluble in hot water. The tests, in fact, united in proving that a disaccharide had been produced. After three weeks, the rotatory power of a 50 per cent solution had fallen to about 16–20 per cent, indicating that roughly 20–25 per cent of the glucose had been converted into disaccharide; this conclusion was confirmed by the change in the reducing power. The isolation of the sugar in a pure state has not yet been effected.

The properties of its phenylosazone resemble those of phenyllactosazone. More light has been thrown on the nature of the sugar by the study of its behaviour towards other enzymes. Having proved that the sugar—which has been termed provisionally *Isolactose*—is unfermentable by "top yeast," the solution, obtained in the manner described, was boiled, diluted, and subjected to fermentation in order to remove the major portion of the unchanged monosaccharides; it was then concentrated, and its behaviour studied towards "bottom" yeast, emulsin, and kefir lactase. All the usual precautions were observed, the extent to which changes took place in the solution was ascertained by determining its reducing power and its rotatory power, when possible, and especially by means of the phenylhydrazine test.

In this way it was proved that, whilst isolactose is entirely fermented by "bottom yeast," and hydrolysed by lactase, emulsin is without any action upon it. Tabulating the behaviour of the natural and synthetic sugars, it is obvious that the two are distinguished by their behaviour towards bottom yeast and emulsin; their relationship is, therefore, of the same order as that which obtains between the α - and β -alkyl-glucosides.

Milk-sugar:—	Top yeast	..	..	Unchanged
	Bottom yeast	..	..	Unchanged
	Emulsin	..	..	Hydrolysed
	Lactase	..	..	Hydrolysed
Isolactose:—	Top yeast	..	..	Unchanged
	Bottom yeast	..	..	Fermented
	Emulsin	..	..	Unchanged
	Lactase	..	..	Hydrolysed

It is, at any rate, certain that milk-sugar is not formed, so that in the case of lactase, as in that of maltase, a sugar isomeric with that normally hydrolysed by the enzyme is obtained as the product of its synthetic activity.

Lactase also exercises a reversible action, though to a much less extent, in a concentrated solution of glucose. In this case, also, a disaccharide is formed which is characterised by yielding a very soluble phenylosazone. On the other hand, little or no synthetical action is effected by lactase in a solution of galactose.

Finally, experiments carried out in an exactly similar manner with *emulsin* show that this enzyme is also capable of effecting synthetical changes, and that it acts in a particularly rapid manner. The maximum of reversion is effected in a concentrated (50 per cent) solution containing equal parts of glucose and galactose, an equilibrium being arrived at when about 20 per cent of disaccharide is present. Reversion also takes place in a concentrated solution of glucose under the influence of emulsin. The products afford phenylosazones which are easily soluble in hot water, and are not fermented by top yeast, but easily by bottom yeast.

It may, therefore, be regarded as established that the enzymes which induce the hydrolysis of glucosides are also capable of acting reversibly in concentrated solutions of the glucoses; the disaccharides which are thus produced, however, are isomeric, not identical with those normally hydrolysed by the enzymes, and in this respect, therefore, the condensing action of enzymes is comparable with that of concentrated solutions of mineral acids.

RECENT SYNTHETICAL RESEARCHES IN THE GLUCOSIDE GROUP.*

By Dr. E. FRANKLAND ARMSTRONG.

If anhydrous hydrogen chloride or bromide be allowed to act at ordinary temperatures on either of the two isomeric pentacetyl-derivatives of glucose, the acetyl radicle attached to the aldehyde group is quantitatively displaced by halogen, giving rise to isomeric aceto-halogen-glucoses, which are well-defined crystalline substances. These compounds are of considerable value as synthetical agents. One of them was made use of by Michael some twenty years ago, in the form of a syrup, in effecting the synthesis of helicin and some other glucosides derived from the phenols. In practice the interaction between the halohydrate and the acetyl-derivative is carried out in sealed tubes, the gases being liquefied by the aid of liquid air. When the interaction is at an end, the tubes are again immersed in liquid air in order to allow of their being opened safely. The aceto-halogen-glucoses are completely converted into the alkylglucosides by treatment with alcohols, giving rise, when methyl alcohol is used, to the known α - and β -methylglucosides.

As the glucosides are obtained in a pure state by this method they crystallise without much difficulty. The β -derivatives are specially easy to obtain, whereas when Fischer's method is used, which involves heating a mixture of the sugar and alcohol together with a small quantity of chlorhydric acid, the greater part of the product is the α -glucoside; the methods therefore supplement one another. A series of alkyl derivatives of glucose and of galactose have been investigated.

Aceto-halogen-derivatives of both maltose and milk-sugar have been prepared, and the former converted into the corresponding β methylmaltoside—the simplest case of a trisaccharide. The behaviour of this compound towards enzymes is specially interesting. As Fischer has shown, maltose is an α -glucoside,† but in the synthetic

* Read before the British Association (Section B), Belfast Meeting, 1902.

† α -Glucosides are those hydrolysed by maltase, β -glucosides those hydrolysed by emulsin.

composed the methoxy-group is introduced into the β -position. Methylmaltoide is, in fact, a double $\alpha\beta$ -glucoside similar to the natural glucoside amygdalin, and resembles this latter in its behaviour with yeast maltase, the maltose part of the molecule being hydrolysed, so that methylglucoside and glucose are formed; on the other hand, when subjected to the action of emulsin, the maltoside is resolved into methyl alcohol and maltose. An exactly analogous behaviour is shown by the corresponding phenolmaltoide.

Recently, β -acetochloro-derivatives of sugars have been prepared by Skraup by the action of phosphorus pentachloride and aluminium chloride on the pentacetyl-derivatives; but in this case molecular rearrangement takes place, both the α - and β -pentacetates giving the same chloro-derivatives. The investigation carried out by the author has shown that the β -halogen-derivatives are the more stable, and that in the presence of alkali the α - are rapidly converted into the β -derivatives, which is a definite proof that the isomerism and likewise that of the glucosides is stereochemical. This instability has unfortunately prevented a synthesis of the α -glucosides of the phenols; and it is of interest to note that nearly all the natural glucosides, which have been tested physiologically, belong to the β -series.

Glucosides of the type of milk-sugar and maltose have been prepared by the interaction of the aceto-halogen-glucoses and the sodium derivatives of the glucoses, and have been isolated in the form of phenylosazones; these osazones are soluble in boiling water and they are converted by the action of benzaldehyde into keto-aldehydes or *osones*, which resemble glucosone in their properties. The disaccharide obtained from acetochlorogalactose and sodium glucose very closely resembles, if it is not identical with, melibiose—the phenylosazones and bromphenylosazones prepared from the natural and synthetic products being indistinguishable, as well as the behaviour of the two sugars towards enzymes. It is noteworthy that the osones derived from the disaccharides resemble the parent sugars in their behaviour towards enzymes and that they, too, exercise a highly selective action.

THE ACTION OF DISTILLED WATER UPON LEAD.*

By Professor FRANK CLOWES, D.Sc.

WHEN lead is placed in contact with ordinary distilled water, chemical action becomes evident by the formation of a *white deposit* which contains lead, and lead is also found in *solution* by chemical tests.

The lead appears to exist *in solution* in the form of hydroxide, since it is removed by passing the liquid through filter-paper; this fixation by cellulose of the dissolved hydroxide is one of the marked properties of the hydroxide. The hydroxide is readily removed from the cellulose by dilute acetic acid.

An examination of the *white deposit* proved that it was a hydroxycarbonate, containing three molecules of carbonate to one of hydroxide.

It would be inferred from the above facts that the lead is probably converted into hydroxide by the action of the oxygen dissolved in the water, and that the subsequent action of the dissolved carbon dioxide converts the hydroxide into hydroxycarbonate.

It would appear improbable that even bright lead can decompose water with formation of hydroxide. But the opinion has prevailed that the action of distilled water on lead is promoted by the presence of carbonic acid.

The nature of the action of distilled water upon lead was studied by exposing bright lead, both *in vacuo* and in

an atmosphere of hydrogen, to distilled water which had been freed from its dissolved gas by boiling; and then introducing either oxygen alone, carbon dioxide alone, or mixtures in varying proportions of oxygen with carbon dioxide.

Bright sheet lead of great purity, when exposed to boiled distilled water *in vacuo* or in an atmosphere of hydrogen, was acted upon only to an infinitesimal extent (0.3 part of lead to one million parts of water). This result was probably due to the impossibility of removing the last traces of oxygen, and it negatives the suggestion of any direct action of water upon lead.

When the lead was exposed to the water in the presence of the different artificial atmospheres, the action reached its greatest extent (0.023 part of lead per 100 of water) when oxygen alone was present; with carbon dioxide the action was slight (0.008 per cent); with equal volumes of oxygen and carbon dioxide about an equal effect was produced; while with eight volumes of oxygen to one of carbon dioxide the extent of the action approached that due to oxygen.

It is evident, therefore, that dissolved oxygen is the cause of the action of distilled water upon lead, the subsequent action of carbonic acid leading to production of hydroxycarbonate. It is further evident that the presence of carbon dioxide in any large proportion exerts an inhibitory effect upon the action.

When sheet lead is immersed in ordinary distilled water with exposure to air, it is found that the rate of action is diminished by total immersion as compared with partial immersion, but that in both cases the total result produced is ultimately the same.

In the earlier stage of the above inquiry the ordinary aerated distilled water was freed from its dissolved gases by being boiled in glass vessels. When this water was allowed to re-aerate itself by exposure to the air, it did not regain its original power of acting upon lead. This was found to be due to the inhibitive power of silicates dissolved from the glass, and the inhibitory power varied with the degree of solubility of the glass, when vessels composed of different kinds of glass were used. Boiling the water in glass, or condensing the steam in glass tube-condensers, lead to the inhibitory effect; but contact of cold distilled water with cold glass did not produce the result. Complications arising from this cause were avoided in later experiments by distilling the water from a copper vessel and condensing the steam in a copper tube-condenser.

It has been suggested that the action of water on lead is indirectly due to the presence of bacteria. In the above experiments, water sterilised by long boiling was frequently exposed to lead which had been fused in a flask sterilised by steam in the presence of air which had passed through cotton-wool; the oxidising action occurred freely in all such experiments, presumably in the absence of bacteria and their products.

The action of distilled water, or of soft waters generally, upon lead may be considerably reduced by dissolving various substances in the water. Sulphuric acid, or a sulphate, was found to be most efficient for this purpose; carbonic acid and carbonates proved less efficient; lime-water was still less effective, and even promoted the action when it was used in larger proportion.

Business Illustrated: an Unconventional Magazine-review. Vol. I, No. 1, July, 1902.—The especial function which *Business Illustrated* has set itself to fulfil is the useful but unconventional one of supplying business people with a publication which stands midway between the trade and technical journals on the one hand and the popular magazine on the other. The new periodical is well and chattily written, and printed on thick glazed paper, while the illustrations are all that can be desired. We wish our young contemporary every success.

* Read before the British Association (Section B), Belfast Meeting, 1902.

THE RADIO-ACTIVITY OF THORIUM COMPOUNDS.*

II. THE CAUSE AND NATURE OF RADIO-ACTIVITY.

By E. RUTHERFORD, M.A., D.Sc.,
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and
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(Concluded from p. 135).

10. *The Nature of the Radiations from Thorium and ThX.*
It has recently been found (Rutherford and Grier, *Phys. Zeit.*, 1902) that thorium compounds, in addition to a type of easily absorbed Röntgen rays non-deviable in the magnetic field, emit also rays of a very penetrating character deviable in the magnetic field. The latter are therefore similar to cathode rays, which are known to consist of material particles travelling with a velocity approaching that of light. But thorium, in comparison to uranium and radium, emits a much smaller proportion of deviable radiation.

From the view of radio-activity put forward, it necessarily follows that the total radio-activity of thorium is altered neither in character nor amount by chemical treatment. This conclusion can be tested by comparing the radiations of thorium and ThX with the mixture which constitutes the thorium radiation. It must, however, be pointed out that it is difficult to make any absolute measure of radio-activity, on account of the different extents in different cases to which the radiations are absorbed in the material of the substance emitting them. The total radio-activity of the original thorium is derived from a small quantity of the substance in the form of powder, while the radiations from ThX are produced by a very thin film of the material on a platinum dish. The radiation from thorium is half absorbed by a thickness of aluminium 0.0004 c.m. thick, and as thorium oxide is far denser than aluminium, it is probable that the radiation in this case is confined to a surface layer only 0.0001 c.m. deep. In the ThX, on the other hand, there is probably but little absorbed in the substance itself. The difficulty can be overcome to some extent by taking for the comparison the radio-activity of a thin film of a soluble thorium salt produced by evaporation; a solution to dryness over a large metal plate. Compared in this way, the radio-activity of ThX when first separated almost exactly equals the activity of the nitrate from which it is produced, while the hydroxide retains about two-fifths of this amount. The difference is in the expected direction, for it is certain that more absorption takes place in the nitrate than in the products into which it is separated. The requirements of the hypothesis can thus be said to be satisfied, but the example illustrates the difficulty of making absolute measurements of radio activity. These throughout have almost completely been avoided. It is possible to trace with great accuracy the change of radio-activity of any preparation by leaving it undisturbed on its original plate, and comparing it always with the same comparison sample. But to express the radio-activity of one body like ThX in terms of that of another like ThX, except for the purposes of comparison, is misleading, as the above consideration shows.

Similar difficulties stand in the way of an answer to the second question—whether the nature of the radiations is affected by chemical treatment,—for it has been experimentally observed that the penetration power of these radiations decreases with the thickness of material traversed. The character of the radiations from ThX and thorium have, however, been compared by the method of penetration power. A large number of comparisons justifies the view that the character of thorium radio-activity is unaltered by chemical treatment and the separation of ThX, although the different types are unequally distributed among the separated products.

* Communicated by the Authors.

The determination of the proportion between the deviable and non-deviable rays affords a new means of approaching the question. The general result is that the radiations from ThX and the excited radiation it produces both comprise deviable and non-deviable radiation. But in the experiment in which the excited radiation was allowed to spontaneously decay—by removing the ThX as formed—the final product, after twenty-three precipitations, was found to be quite free from deviable radiation. This, as will be shown in a following paper, is one of the most striking resemblances between the non-separable radio-activities of uranium and thorium.

Finally, it may be mentioned that the proportion of deviable and non-deviable radiation is different for different compounds of thorium. The nitrate and ignited oxide, compounds which hardly possess any emanating power, have a higher proportion of deviable radiation than compounds with great emanating power. This is indirect evidence of the correctness of the view already put forward (Section 7), that when the emanation is prevented from escaping, it augments the proportion of excited radio-activity of the compound.

11. Summary of Results.

The foregoing experimental results may be briefly summarised. The major part of the radio-activity of thorium—ordinarily about 54 per cent—is due to a non-thorium type of matter (ThX) possessing distinct chemical properties, which is temporarily radio-active; its activity falling to half value in about four days. The constant radio-activity of thorium is maintained by the production of this material at a constant rate. Both the rate of production of the new material and the rate of decay of its activity appear to be independent of the physical and chemical condition of the system. The ThX is undergoing a further change, and one of the products is gaseous, and in the radio-active state constitutes the emanation produced by thorium compounds. The ThX further possesses the property of exciting radio-activity on surrounding inactive matter, and about 21 per cent of the total activity under ordinary circumstances is derived from this source. Its rate of decay, and other considerations, make it probable that it is the same as the excited radio-activity produced by the thorium emanation, which has been shown to be produced by ThX. There is evidence that if by any means the emanation is prevented from escaping in the radio-active state, the energy of its radiation goes to augment the proportion of excited radio-activity in the compound.

Thorium can be freed by suitable means from both ThX and the excited radio-activity which the latter produces, and then possesses an activity about 25 per cent of its original value, below which it has not been reduced. This residual radiation consists entirely of rays non-deviable by the magnetic field, whereas the other two components comprise both deviable and non-deviable radiation. Most probably this residual activity is caused by a second non-thorium type of matter produced in the same change as the ThX, and it should therefore prove possible to separate it by chemical methods.

12. General Theoretical Considerations.

Turning from the experimental results to their theoretical interpretation, it is necessary to first consider the generally accepted view of the nature of radio-activity. It is well established that this property is the function of the atom and not of the molecule. Uranium and thorium, to take the most definite cases, possess the property in whatever molecular condition they occur, and the former also in the elementary state. So far as the radio-activity of different compounds of different density and states of division can be compared together, the intensity of the radiation appears to depend only on the quantity of active element present. It is not at all dependent on the source from which the element is derived, or the process of purification to which it has been subjected, provided sufficient time is allowed for the equilibrium point to be

reached. It is not possible to explain the phenomena by the existence of impurities associated with the radio-active elements, even if any advantage could be derived from the assumption. For these impurities must necessarily be present always to the same extent in different specimens derived from the most widely different sources; and, moreover, they must persist, in *unaltered amount*, after the most refined processes of purification. This is contrary to the accepted meaning of the term impurity.

All the most prominent workers in this subject are agreed in considering radio-activity an atomic phenomenon. M. and Mme. Curie, the pioneers in the chemistry of the subject, have recently put forward their views (*Comptes Rendus*, 1902, cxxxiv., p. 85). They state that this idea underlies their whole work from the beginning, and created their methods of research. M. Becquerel, the original discoverer of the property for uranium, in his announcement of the recovery of the activity of the same element after the active constituent had been removed by chemical treatment, points out the significance of the fact that uranium is giving out cathode rays. These, according to the hypothesis of Sir William Crookes and Professor J. J. Thomson, are material particles of mass one-thousandth of the hydrogen atom.

The present researches had as their starting-point the facts that had come to light with regard to the emanation produced by thorium compounds and the property it possesses of exciting radio-activity on surrounding objects. In each case the radio-activity appeared as the manifestation of a *special kind of matter* in minute amount. The emanation behaved in all respects like a gas, and the excited radio-activity it produces as an invisible deposit of intensely active material, independent of the nature of the substance on which it was deposited, and capable of being removed by rubbing or the action of acids.

The position is thus arrived at, that radio-activity is at once an atomic phenomenon and the accompaniment of a chemical change in which new kinds of matter are produced. The two considerations force us to the conclusion that radio-activity is a manifestation of sub-atomic chemical change.

Before such a view was entertained, the possibility of explaining the results obtained on the existing hypotheses was fully considered. But the attempt was unsatisfactory, and involved the assumption of many new conceptions which cannot be experimentally verified, and which are, moreover, not in accordance with what is already known of radio-activity. It is apparent in radio-activity that we are dealing with phenomena outside of the sphere of known atomic forces, for the whole process proceeds independently of temperature and chemical affinity. So that even if the accepted views were altered, and the radio-active elements regarded as compounds that had not yet been resolved, it would not assist in the interpretation of the facts. Besides, there is not the least evidence for assuming that uranium and thorium are not as homogeneous as any other chemical element, in the ordinary sense of the word, so far as the action of *known* forces is concerned. The idea of the chemical atom in certain cases spontaneously breaking up, with evolution of energy, is not of itself contrary to anything that is known of the properties of atoms. For the causes that bring about the disruption are not among those that are yet under our control, whereas the universally accepted idea of the stability of the chemical atom is based solely on the knowledge we possess of the forces at our disposal.

The changes brought to knowledge by radio-activity, although undeniably material and chemical in nature, are of a different order of magnitude from any that have before been dealt with in chemistry. The course of the production of new matter which can be recognised by the electrometer by means of the property of radio-activity after the course of a few hours, or even minutes, might possibly require geological epochs to attain the quantities recognised by the balance. However, the well-defined

chemical properties of both ThX and U₂X are not in accordance with the view that the actual amounts involved are of this extreme order of minuteness. On the other hand, the existence of radio-active elements at all in the earth's crust is an *a priori* argument against the magnitude of the change being anything but small.

It is a significant fact that the radio-active elements are all at the end of the periodic table. If we suppose that radium is the missing second higher homologue of barium, then the known examples—uranium, thorium, radium, polonium (bismuth), and lead—are the five elements of heaviest atomic weight. Nothing can yet be stated of the mechanism of the changes involved; but whatever view is ultimately adopted, it seems not unreasonable to hope that radio-activity affords the means of obtaining information of processes occurring within the chemical atom.

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THE REVERSION OF SUPERPHOSPHATE OF LIME IN THE SOIL.

By W. F. SUTHERST, Ph.D., A.I.C.

SUPERPHOSPHATE of lime is manufactured by the action of strong sulphuric acid on any substance containing a large amount of tricalcic phosphate, such as coprolites, bones, &c., sufficient acid being employed to combine with two-thirds of the total calcium, forming calcium sulphate and monocalcium phosphate, soluble in water. This product has been used with very great success as a manure for a number of years, and at the present time constitutes the chief source of phosphoric acid for fertilising purposes. It is usual to add this manure to the soil in quantities varying from 2 to 5 cwts. per acre, according to the kind of crop grown.

Nearly the whole of the phosphoric acid present in superphosphate is—or should be—soluble in water; when freshly made there is more soluble than on keeping, the monocalcic salt changing slowly to the dicalcic. But when applied to the soil the plants do not take it up in that form, since it has been shown that free acid (especially mineral) kills most plants very quickly, but it first undergoes a change, being rendered insoluble by several ingredients present, the chief ones being carbonates of calcium and magnesium, oxides of iron and aluminium—in fact, any oxide or carbonate of a base which happens to be present. The acid- or water-soluble phosphate, then, when it comes in contact with the soil, combines with these ingredients, forming insoluble calcium, magnesium, or iron phosphates; and it is from these compounds that the plants obtain the phosphoric acid necessary for their growth, the acid sap of their roots being capable of dissolving to some extent these compounds, and by the process of diffusion absorbs them into the plant system.

This process of precipitation or reversion does not by any means take place instantly; many days elapsing before all the water-soluble phosphate is rendered insoluble, and that only when a large excess of the reverting agents are present—a state of things not always occurring.

Should there be only small quantities of chalk or iron oxide present the superphosphate is liable to be washed out of the soil, especially if of a sandy nature; this easily explaining the reason why light sandy soils give such poor results when superphosphate or other soluble fertilisers are applied.

To observe which ingredient of the soil was most active, and also to find how much of each was necessary to revert the soluble phosphate, a series of experiments were carried out by me in the laboratories of the Agricultural College, Holmes Chapel. The experimental part was carried out in the following manner:—In a number of flasks a known weight of superphosphate of known strength was mixed with water, and to this solution various amounts of chalk,

magnesium carbonate, and limonite (the latter representing as near as possible the oxide of iron in the soil), and from these at different periods samples were taken and the phosphoric acid still soluble in water estimated by the ammonium molybdate and magnesia mixture method; most of the estimations being carried out most assiduously by Mr. F. C. Harold, one of my students. The following table gives the amount of each ingredient used to one part of superphosphate, the time taken for reversion, and the amount of phosphoric acid still in solution.

Parts super-phosphate, containing 227 per cent sol. P_2O_5 .	$CaCO_3$ added.	Time taken for reversion.	P_2O_5 still in solution.	Per cent in solution to per cent total P_2O_5 .
	Parts.	Days.		
I	9	15	0.044	19.59
I	12	15	0.040	17.70
I	24	15	0.022	9.70
MgCO₃ added.				
I	9	9	0.014	6.12
I	12	9	0.0024	1.06
I	24	6	0.0019	0.83
Limonite added.				
I	9	9	0.046	20.26
I	12	9	0.038	16.74
I	24	9	0.0091	4.00

From the above results it appears that the magnesium carbonate is the most active reverting agent, the oxide of iron next, and the calcium carbonate the least of all; in each case, however, some portion of the phosphate remained in solution.

The soil generally contains more oxide of iron than any of the other ingredients, except in the case of the soils resting on chalk, so that in most cases the soluble phosphate is precipitated chiefly as an iron compound, the remainder combining with the calcium and magnesium carbonates. Now, it is from these compounds that the root-sap takes up phosphoric acid, but only the calcium and magnesium compounds are taken up (the iron phosphate not being soluble), so that by adding superphosphate to ordinary soils only about one-half is available for plant food. To prove this, mixtures were made up of calcium and magnesium carbonates and limonite, and to these were added a known amount of superphosphate, together with water. After standing some days, the amount of phosphoric acid still in solution was determined, and after this a portion of the contents was treated with 1 per cent citric acid solution (this being Dyer's method of representing the acidity of root-sap), and the phosphoric acid dissolved out estimated. The following table gives the results:—

Mixture.	Time taken for reversion. Days.	P_2O_5 in solution to total P_2O_5 . Per cent.	P_2O_5 soluble in citric acid. Per cent.
1. Representing a clay soil. Containing 10 parts limonite, 4 parts $CaCO_3$, 1 part $MgCO_3$	12	12.64	54.00
2. Representing a loamy soil. 10 parts limonite, 2 parts $CaCO_3$, 1 part $MgCO_3$	12	14.34	50.00
3. Representing a loamy soil. 10 parts limonite, 1.5 parts $CaCO_3$, 20 parts $MgCO_3$	12	14.40	49.40

The neutral or basic phosphatic manures, such as basic slag or basic superphosphate, are in the insoluble form already, and being combined chiefly with calcium, the greater part of the phosphoric acid is thus available for plant food; this being especially the case with basic superphosphate, the whole of whose phosphoric acid is soluble in citric acid solution.

Cheshire Agricultural College,
Holmes Chas. et.

THE SUPPOSED DISCOVERY OF ALUMINIUM TWO THOUSAND YEARS AGO.

THE following letter appears in *Knowledge* for September, 1902:—

SIRs,—The following paragraph appeared in "Echoes of Science" in the *Globe* of August 19th, 1898:—

"The first discoverer of aluminium had the reward of genius. Pliny tells us that in the reign of Tiberius (41 B.C. to 37 A.D.) a worker in metals presented a beautiful metal cup resembling silver, but lighter, to the Emperor, who questioned him, and learned that he had extracted the new metal from clay. The secret, he said, was known but to himself and the gods. The sage Tiberius, reflecting that if this metal could be made from earth it would lower the price of silver and gold, decapitated the artificer in order that his secret might remain with the gods, and so deprived the world of a most useful metal for eighteen centuries."

It would interest many readers of *Knowledge* if some student of Pliny would state where in his works the account is to be found, and furnish a translation thereof.

That the metal of which the cup was formed was really aluminium appears possible from four circumstances:—(1) It was obtained from clay; (2) it resembled silver; (3) it was lighter than silver; (4) it was capable of being wrought into a vessel.

The question naturally occurs, How did the metallurgist of the first century obtain a metal, which in the twentieth century can only be extracted by processes totally unknown to the ancients, and which it is inconceivable that any individual worker among them could have accidentally hit upon? Modern methods of procuring aluminium are:—(1) Chemical, involving the use of sodium or potassium; and (2) electrical. The contemporary of Tiberius cannot have knowingly isolated sodium or potassium, and then applied it to separate aluminium, though it is possible that sodium or potassium may have been liberated from some ingredient of a mixture in his retort or crucible, only to oust aluminium from some other ingredient. That his process was electrical is quite out of the question.

If Pliny's account actually refers to aluminium there must be some method of separating it which modern chemists have failed to light on, but which lay not very far outside of the range of ancient metallurgical knowledge. Cannot it be re-discovered by some chemist possessing a wide knowledge of the science, and who is at the same time well acquainted with the methods (many of them forgotten) adopted by ancient and mediæval workers? Such a process would probably go very far towards cheapening that most useful metal.

JOHN T. KEMP.

Report of "The Lancet" Special Commission on Glycerinated Calf Vaccine Lymphs.—In this report some interesting results of experiments are given concerning the number and nature of the extraneous bacteria found in the commercial vaccine lymphs. We note that there is scarcely a doubt that all the vaccine lymphs at present on the market are, as regards their capability of producing vaccine vesicles, of very trustworthy character at some period or other after their preparation; it is, however, recommended that where any doubt occurs, over-glycerinated rather than under-glycerinated lymph should be used. Many of the cases of bad vaccination are due to either imperfect sterilisation of the skin, or want of protection against the invasion of the weakened and abraded tissues by extraneous organisms. A table giving the results of experiments with sixteen different samples of lymph shows that the numbers of colonies per 0.05 gm. vary from 0 in the case of Merck's lymph to 10,000 in that supplied by Chaumier, and *unaccountable* in the case of that from the "Association for the Supply of Pure Lymph (Darke)" on agar plates.

NOTICES OF BOOKS.

The New Volumes of the Encyclopædia Britannica. The Fifth of the New Volumes, being Vol. XXIX. of the Complete Work. London: *The Times*. Edinburgh: Adam and Charles Black. 1902. Pp. 763.

THE subjects dealt with in this volume of the Encyclopædia, are those comprised between the alphabetical headings G L A (Glarus) and J U T (Jutland).

The first article to attract attention is one on "Glass," by Mr. H. J. Powell. Manufactured glass is classified under three principal headings:—I., Optical Glass; II., Blown Glass; III., Glass Pressed or Blown Mechanically. The author draws attention to the deterioration in the quality of glass articles, owing to the demand by the public for regularity. He says:—

"The beauty of opal glass consists in the wanton irregularity of its shading, no two pieces being precisely similar. The public asked for regularity, and have been supplied with an insipid milk and water material which is called opal. 'Threading' has suffered much in the same way. . . . A lathe has been introduced which not only winds the glass thread with exasperating exactitude, but renders it possible to cover a vessel with threading from top to bottom."

Mr. J. Hays Hammond contributes a short article on Gold, consisting principally of statistics.

The article on Hygiene, by Col. J. Lane Nottle, though not very long, is well written and interesting; in it he touches on the influence of soil in the production of disease, the purification of sewage by means of biological treatment, the purification and sterilisation of water, quarantine, plagues, and epidemics.

"Iron and Steel" forms the subject of an important article by Prof. H. M. Howe; the author points out that during the last twenty years the world's production of pig-iron has been more than doubled, and that of steel increased five-fold, the greatest increase being in the United States. Delicate methods of research have revealed the nature and constitution of several varieties of iron; this knowledge has contributed in no small degree to the important beginning which has been made in the use of rational methods of thermal treatment, and it has aided in the discovery and utilisation of numerous "alloy" steels. This article is well illustrated with modern appliances for iron and steel making.

International Catalogue of Scientific Literature. First Annual Issue. D. Chemistry. Vol. II., Part I. Published for the International Council by the Royal Society of London. London: Harrison and Sons. 1902 (June). Pp. 468.

THE possibility of preparing a complete index of current scientific literature by international co-operation was first taken into consideration by the Royal Society about the year 1893, and the Society therefore sought the opinion of a very large number of representative bodies and individuals abroad, the result being that a Conference took place in London on July 14-17, 1896, at which it was unanimously resolved to compile and publish a complete Catalogue of Scientific Literature, arranged according both to subject-matter and author's names, in which regard should be had, in the first instance, to the requirements of scientific investigators, so that these might find out, with a minimum of trouble, what had been published on any particular subject of enquiry.

The supreme control over the Catalogue is vested in an International Convention to be held in London in 1905, 1910, and every tenth year afterwards.

There are seventeen branches of science to be included in the Catalogue, and thus each complete annual issue of the Catalogue will consist of seventeen volumes, and a schedule of classification and an index thereto will be prefixed to each volume in English, French, German, and Italian. The various headings and sub-headings through-

out the subject-index are given in English, and translations of the main headings can be found on reference to the schedules in the other languages by means of the registration numbers that are attached to them.

In the Author's Catalogue each title is given in the original language.

The present volume consists of three parts:—(a) Schedules and Indexes in four languages; (b) An Author's Catalogue; (c) A Subject Catalogue.

The Subject Catalogue is divided into sections, each of which is denoted by a four-figure number between 0000 and 9999. These numbers follow each other in numerical order, but all are not used, as it is intended to fill up the gaps by interpolation of such additional sections as may be required in future years. At the end of the volume a large number of the organic substances referred to in the Subject Catalogue are arranged in alphabetical order; the four-figure numbers given in this list are the registration numbers. In the Author's Catalogue the four-figure numbers in square brackets at the end of each entry are registration numbers.

A large amount of care and thought has been devoted to the compilation of this Catalogue, and it cannot fail to be of great value to scientific men all the world over.

Suggested Standards of Purity for Foods and Drugs. By C. G. MOOR, M.A., F.I.C., F.C.S. London: Baillière, Tindall, and Cox. 1902. Pp. 260.

MANY years ago an attempt was made by the Society of Public Analysts to suggest certain limits or standards as indicating the genuineness of certain articles, such as milk, butter, &c. Some of the figures then suggested are still in use, and the aim of the present work is to endeavour to extend the same principle to other articles that come within the scope of analysts' duties generally, but the figures here suggested must be accepted as tentative only.

With regard to medicinal preparations, the original intention was to deal with all British Pharmacopœial preparations, but as this proved too large a task, a selection had to be made, which led to the omission of most chemicals which either are not likely to be adulterated or for which there are definite chemical tests offering no difficulties.

In considering the merits of any particular article, due regard must be had to the purpose for which it is intended, as there may be a legitimate use for an article which need not be pure in certain cases, whereas if intended for medicinal use its purity would be essential. The book has been carefully compiled, and will be of great assistance to analysts in general.

Hilfsbuch zur Ausführung Chemischer Arbeiten. ("Aids to Practical Chemistry"). By HUGO SCHWANERT. Braunschweig: Friedrich Vieweg und Sohn. 1902.

THIS fourth edition of Dr. Schwanert's "Hilfsbuch" has been considerably enlarged, new methods of separation and determination having been added, while the general arrangement is unaltered. The analysis of inorganic and organic mixtures is very fully treated, and the tables for systematic procedure are particularly good. The section on the preparation of various chemical substances also will be found very useful, the directions being both concise and complete; it might perhaps have been preferable to open the book with this section, deferring the systematic analysis until later. The most important and commonly used methods of gravimetric and volumetric analysis are given (in the latter case very fully); and, finally, the book contains an introduction, by no means superficial, to the analysis of water, food materials, and poisons, together with some experiments in zoo-chemistry, such as the examination of the animal secretions, blood, horn, &c. In the case of milk analysis, the Werner-Schmid method for determining fat is not recommended, although it has been found in practice to be both rapid and accurate.

The General Principles of Physical Science. By ARTHUR A. NOYES. New York: Henry Holt and Co. 1902.

THE author of this book has not succeeded in producing an attractive or eminently readable treatise on the subjects he deals with. Though the style is, on the whole, clear, some parts are decidedly dull, and unlikely to stimulate the reader's interest. Occasionally, statements which are really misleading are made. For instance, the paragraph on specific gravity and density may be cited as likely to lead to confusion in the mind of the student, especially if he had no previous knowledge of the subject. The chapter on the General Principles relating to Matter provides a short treatment of the theory of chemistry; and that on the General Principles relating to Energy, a course of the theory of physics, the second law of energetics being fully discussed. The treatment throughout professes to be entirely non-mathematical, but as some knowledge of the Calculus is assumed, this will probably be of no very great advantage to the ordinary student.

work was dissolved in a liquid metal under great pressure, and cooled. The carbon in my work was also in contact with—and probably dissolved in—liquid metal under great pressure and then cooled. The metal in Moissan's case was iron, and in mine it was potassium and iron; and although my temperature may have been lower, I had proved that there was a violent reaction between the potassium and the contents of the tube, which would raise the temperature locally to as high as Moissan's, and so produce identical conditions, as the carbon was in contact with the iron.

The experiment was planned from discoveries made in my research into the "Solubility of Solids in Gases," and Moissan quotes my paper as the first successful production of crystalline carbon in the diamond form.

Apologising for the length of my explanation.—I am, &c.,

J. B. HANNAY.

Cove Castle, Loch Long, N.B.

CORRESPONDENCE.

ATOMIC WEIGHT STANDARDS AND PROUT'S HYPOTHESIS.

To the Editor of the Chemical News.

SIR,—In the CHEMICAL NEWS for September 19 (vol. lxxxvi., p. 147), Mr. Hollins puts forward a view upon the integral nature of atomic weights analogous to that of Prout; but unlike Prout, who made but a superficial generalisation, Mr. Hollins would endeavour to uphold his view by figures calculated to five places of decimals.

To a practical chemist this appears "false accuracy." In the present uncertainty of many atomic weights—even those of the common elements—it appears quite unreasonable to make use of figures taken out to five places when dependent upon figures in which uncertainty appears at least in the second place, and frequently in the integers themselves. There has been a tendency for many atomic weights to fall—as much as two whole numbers in some cases—during the last few years, and we are by no means sure that the numbers now current are final; consequently, it would appear more scientific to make use of figures involving the first place of decimals only. The hypothesis of Prout has at best but doubtful value and bearing upon physical and chemical science, and certainly this value is not enhanced by the use of figures of such fictitious accuracy in its proof.—I am, &c.,

GEO. H. WOOLLATT.

11, Old Park Avenue, Clapham Common, S.W.,
September 22, 1902.

PRODUCTION OF DIAMOND.

To the Editor of the Chemical News.

SIR,—Will you kindly allow me to make a note in your valuable paper on the statement, in the new volumes of the "Encyclopædia Britannica," that the hard substance I found in my tubes was really carborundum (article, "Gems, Artificial")?

Had the original paper or the correspondence in the *Times* been consulted, this could hardly be asserted, as it was fully tested at the time, and was found to be converted entirely into carbon dioxide when burnt in air or oxygen.

But putting that aside, and even the fact that no silicon was used in my experiments, if we accept as a fact (and we all do) that Moissan's carbon was true diamond, then, as the material I made was produced under nearly identical conditions, it is clear that Moissan's experiments really corroborate my result. The carbon in Moissan's

PROPOSED METHOD FOR DETERMINING THE GENUINENESS OF MILK.

To the Editor of the Chemical News.

SIR,—No method, so far as I know, has hitherto been suggested for the discrimination of a milk naturally poor in butter-fat from a rich milk reduced by the addition of separated milk, &c. The obvious advantages that would accrue from the possession of such a method must be my excuse for trespassing on your space at this time with a preliminary note on the result of certain observations and experiments which promise to give assistance in this direction.

Early in my practice as a milk analyst I noticed a seasonal variation in the fatty contents of milk, which reached its maximum in October or November, and had a minimum value about the month of May. This has been recorded by many other observers, and is now well known among those interested in dairy matters.

In the course of similar observations on the composition of butter, I came on a like seasonal variation in the proportion of volatile fatty acids, but having its maximum and minimum points in portions of the year opposite to those in which the maximum and minimum of fat in milk occur.

This variation has been lately put on record by a Dutch observer, whose book was noticed in the CHEMICAL NEWS a short time ago.

Though it is some time since the possibility of a connection between these two phenomena suggested itself to me, it was only lately I had leisure to make the following experiments with a view to finding whether there was any difference as regards volatile fatty acids between the fat of naturally rich and naturally poor milk in favour of the latter. The most crucial case seemed to be that of the morning and evening milk of the same cow, between which there is generally a well-marked difference. Through the kindness of a friend, I obtained a quart each of morning and evening milk from one cow. The cow had calved three months before, and gave in the morning (at 6 a.m.) 2 gallons 4½ pints, and in the evening (at 3 p.m.) 1 gallon 4½ pints. The proportions of fat were—3·20 per cent in the morning and 3·80 per cent in the evening. The two samples were allowed to rest, the greater part of the skim milk drawn off with a syphon, and the cream shaken in the bottle till butter had formed. On estimating the volatile fatty acids in the usual way, 2·5 grms. of fat from the morning milk gave Reichert figures of 14·80 c.c. and 14·85 c.c., while that from the portion naturally richer in fat gave only 14·10 and 14·00.

From this it would appear that there is a distinct *inverse* relation between the quantity of naturally occurring fat and that of the volatile fatty acids, though the one variation does not completely counterbalance the other; the

fat in the evening milk being only one-twentieth poorer in volatile fatty acids, while one-fifth greater in quantity. Further investigation may, however, furnish sufficient data to enable an opinion to be formed in certain doubtful cases. If, for instance a sample of milk was found poor in fat, and yet the fat was richer in volatile acids than the average for the particular season, this fact would furnish evidence of the genuineness of the milk, for the fat of a rich milk diluted to the same strength would be found to contain less than the average amount of volatile acids.

I trust that dairy chemists who have the opportunity may follow up this question in its various branches.—I am, &c.,

THOS. FARRINGTON, M.A., F.C.S., F.I.C.,
Analyst to the County of Cork
Agricultural Society.

Cork, September 25, 1902.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Seances, de l'Académie des Sciences. Vol. cxxv., Nos. 7 and 8, August 19 and 26, 1902.

These two numbers contain no chemical matter.

No. 9, September 1.

Action of Soluble Ferments and of Yeast on Gentianose. Remarks on the Constitution of Gentianose.—Em. Bourquelot and H. Hérissey.—Up to the present time only two polysaccharides were known which were attacked by invertine; these were saccharose and raffinose. Gentianose is now found to form a third, and recently M. Tanret found a fourth—mannetotrose. In the four cases one molecule of levulose is broken up. The phenomenon has a general aspect, and it seems that it is possible to define it thus:—Only the polysaccharides containing one molecule of levulose united to one molecule of glucose in the same manner as saccharose are attacked by invertine, and that with breaking up of the levulose. To completely hydrolyse gentianose, and probably all the polysaccharides, several ferments are necessary. For a body composed of three molecules there must be two which are invertine and emulsine, or at least a ferment contained in the emulsine of almonds. Further, our experiments show that the actions of fermentation are not simultaneous, that of invertine preceding that of emulsine, and since that which hydrolyses biose is without action on triose.

No. 10, September 8.

A New Acidimetric Indicator.—L. J. Simon.—Amongst the products which are formed as an accessory during the calcination of tartaric acid in presence of potassium bisulphate is a new compound, $C_7H_5O_3$, isomeric with pyrotritaric acid, and to which the author assigns the name isopyrotritaric acid. Ferric solutions communicate to an aqueous solution of this an intense violet colouration. This colour is apparently due to the formation of iron isopyrotritarate, a well-defined crystalline compound, $(C_7H_5O_3)_3Fe \cdot 2H_2O$, which serves as an indicator in acidimetric measurements. It has the curious property of giving indications which are usually obtained by the successive use of helianthine and phenolphthalein. The salt, which is very soluble in water, gives it a brownish red tint, nearly black in concentrated solution. On dilution, it becomes orange-red, then reddish yellow. Acids turn this colour to violet in concentrated solution, and to rose in dilute solution. Alkalis, on the contrary, cause a decolouration of the solution to a pale yellow tint.

MISCELLANEOUS.

The South-Western Polytechnic, Chelsea.—This Institute is divided into two distinct portions, day classes and evening classes. The day classes comprise a college for men, a college for women, and a day school for boys and girls. The college for men is at present attended by more than 150 students, whose ages run from sixteen to forty. The courses are arranged to occupy three years. On entering, the student will state whether he wishes to be trained as a mechanical, civil, or electrical engineer, or as a consulting or industrial chemist. In any of these cases he will find mapped out for him a complete course of study, involving laboratory instruction, tutorial work, attendance at lectures, exercises in mathematics, geometrical, mechanical, and architectural drawing, and instruction in the workshops. It is necessary that those who enter for technical instruction should have received previously a sound English education, and have acquired an elementary knowledge of mathematics, and, if possible, of physics and chemistry. The objects of the evening classes are to offer to artisans and others, both men and women, engaged in technical and commercial industries, the means of instruction in applied science and art, and also to women opportunities of training in cookery, dressmaking, and household management; to afford instruction which will be of service to those who intend to enter on a Colonial life, &c. The classes and lectures in technical subjects are designed to supplement, and not to supersede, the training of the workshop.

NOTES AND QUERIES.

Society of Electro-chemists.—Could you inform me if there is in London a Society (or Club) of Electro-chemists, and, if so, where their meetings and reunions are held? Could you also advise me whether there is in London any institution where German chemical papers can be read? I shall be greatly obliged for any information you can give me on these subjects.—Dr. MAX REUTER.

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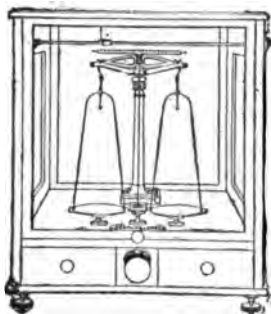
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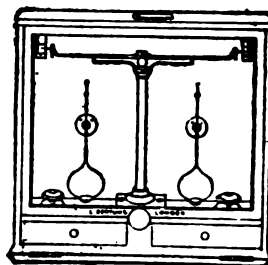


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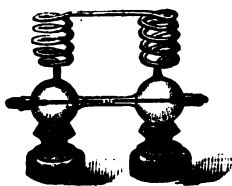
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THE CHEMICAL NEWS.

Vol. LXXXVI., No. 2337.

THE DECOMPOSITION OF UREA.*

By C. E. FAWSITT, Ph.D.

THE author has studied the decomposition of urea into ammonia and carbonic anhydride in aqueous solution at 99° C. as a problem in reaction velocity. The reaction in presence of either acid or alkali would be expected theoretically to be bi- or tri-molecular, but the experimental results show that in these cases, and also when pure water is used as the solvent, decomposition proceeds in accordance with the formula of a mono-molecular reaction. This apparent anomaly is explained by assuming the formation of an intermediate product, namely, of ammonium cyanate; further, the presence of this salt in the solution, as an intermediate product and not as a by-product, has been proved.

COLLOIDS OF ZIRCONIUM, COMPARED WITH THOSE OF OTHER METALS OF THE FOURTH GROUP.*

By Dr. J. H. GLADSTONE, F.R.S.,
and
WALTER HIBBERT, F.I.C.

ON continuing the experiments on colloids, which were described in their papers at Dover and Glasgow, the

that have previously been observed in solutions of silicon. All these colloids seem to be marked by the extreme readiness with which the hydroxyl element replaces more or less chlorine, and by the number of molecules of water with which they will combine.

ISOMORPHOUS SULPHONIC DERIVATIVES OF BENZENE.*

THE crystallographic study of the 1:3:5 series, the *second* of the three series of sulphonic chlorides and bromides to be derived from the 1:3-dichloro-, chlorobromo-, and dibromo-benzenes examined thus far, has been practically completed by Dr. Jee during the past year. The results are even more striking than those obtained in the case of the 1:3:4-series, as there is evidence that the compounds constitute not merely an *isotrimorphous* group, as in the latter case, but an *isotetramorphous* group, as shown in the accompanying table.

In this series the dibromobenzenesulphobromide has again been the means of formulating the relationships between the various terms, having been obtained in two distinct polymorphic forms. But in comparison with the 1:3:4 series the order of stability is reversed, the transition temperature becoming lower in passing down the series.

It is proposed to submit an account of the results obtained in the case of the 1:4:2(SO₃H), 1:3:4(SO₃H), and 1:3:5(SO₃H) series to the Royal Society in the autumn.

	Orientation.			Crystallographic systems.			
	1.	3.	5.	Anorthic.	Monosym.	Monosym.	Anorthic.
1.	Cl	Cl	SO ₂ Br	Stable	Stable	—	—
2.	Cl	Br	SO ₂ Br	—	Stable	—	—
3.	Br	Br	SO ₂ Br	—	Stable	Labile	—
4.	Br	Br	SO ₂ Cl	—	—	Stable	—
5.	Br	Cl	SO ₂ Cl	—	—	Stable	—
6.	Cl	Cl	SO ₂ Cl	—	—	—	Stable
				α-Series	β-Series	γ-Series	δ-Series

authors found that zirconium gave a colloid of well-marked properties, resembling those of silicon, tin, titanium, and thorium. The tetrachloride of zirconium is a volatile pungent body which, reacting with water, forms hydrochloric acid and various oxychlorides or hydroxides. The gummy mass so obtained, when dried, gave beautifully regular forms of fissures and spirals. The gummy solution, when heated, formed a gel which, when exposed to the air, gradually lost water. Successive diffusates from this gel gave various crystalline forms on evaporation. The first diffusate was particularly rich in dagger-shaped crystals with rounded edges, which proved to be oxychlorides. In later diffusates this compound was gradually replaced by crystals of other forms, similar to what had been previously observed among the colloids of tin and titanium, and which appeared to be free from chlorine. These crystals were extremely labile, and exhibited forms always more or less curved in their outlines. The solutions of these colloids of zirconium, tin, and titanium slowly formed crusts or skins which were insoluble in water, and which slowly shrank, often cracking in irregular patterns. It is a curious circumstance that the solution of zirconium colloid very readily produces spongy growths, similar in appearance to those

The third series of the 1:3 derivatives, in which the sulphonic group is between the halogens, has yet to be examined. Very great difficulty has been experienced in preparing the necessary material, but methods have now been devised which promise success. Thus diorthobromobenzenesulphonic acid has been prepared from diorthobromaniline by Gattermann's sulphinic acid method. Unfortunately, the corresponding dichloro- and chlorobromaniline are not at present available, and therefore other methods of preparing the dichloro- and chlorobromacid need to be devised. Apparently these will be obtainable from the dimeta-derivatives of aniline.

A complete series of 1:2:4(SO₃H) derivatives has been prepared, and their crystallographic study will now be undertaken. To complete the investigation, so that it shall comprise all the series derivable from the dichloro-, dibromo-, and chlorobromo-benzenes, only the 1:2:3(SO₃H) series has to be prepared. This, again, is a matter of considerable difficulty, requiring special investigation.

Progress has been made in extending the investigation to the comparison of the effect produced by homologous hydrocarbon radicles, and also in contrasting corresponding sulphur and oxygen compounds.

* Read before the British Association (Section B), Belfast Meeting, 1902.

* Third Report of the Committee, consisting of Professor H. A. Miers (Chairman), Dr. W. P. Wynne, Mr. W. J. Pope, and Dr. H. E. Armstrong (Secretary). Drawn up by the Secretary. Read before the British Association (Section B), Belfast Meeting, 1902.

ON FLUORESCENT AND PHOSPHORESCENT
DIAMONDS.*

By Dr. J. H. GLADSTONE, F.R.S.

In the *Comptes Rendus* of the French Academy of May 20 last there is a short communication by M. Chaumet on the action of light on precious stones, in which he describes the fluorescence excited in some diamonds by violet light, and mentions the case of a yellow diamond which showed no fluorescence under those rays, but after a few minutes' exposure became dark brown; after twenty-four hours, however, its original colour and brilliancy returned.

This reminded me of the diamond ring which I exhibited at the meeting of the British Association at Aberdeen in 1859, containing five stones, two of which were strongly fluorescent in sunshine and one slightly so, while the other two did not exhibit that property. These fluorescent diamonds continued to phosphoresce for some hours in the dark (see *Brit. Assoc. Report*, 1859, Trans. Section B, p. 69). The phenomenon was afterwards exhibited by the Rev. Dr. Robinson at the evening lecture. A few days later, when on a visit to a friend who had a remarkably fine collection of jewellery, I had the opportunity of examining her diamonds in the sunshine, but not one of them showed the least trace of fluorescence. I concluded, therefore, that diamonds of the first water did not possess this property. Possibly the fluorescence in the three diamonds of the ring may have depended upon some impurity. It was found subsequently that they shone most brightly when exposed to the violet and ultra-violet rays. A long exposure to sunlight, however, exhausted the power of phosphorescence for a time, but this was not due to any increase of temperature.

In 1896, Prof. Silvanus Thompson exhibited the ring during the course of his lecture on electric shadows and luminescence at the Royal Institution. The diamonds shone very brilliantly, but next day it appeared they had entirely lost their phosphorescent power. They were kept in the dark, and when examined a few months afterwards it was found that the power of phosphorescing was restored, though not to its former extent.

M. Chaumet says that all diamonds, whatever their quality, exhibit the same transparency to the X-rays. I happen to possess a radiograph of the ring, taken in January, 1897; it shows a slight amount of absorption by all the five diamonds, in strong contrast to the great absorption of the gold setting; and there is no discernible difference between the diamonds that fluoresce and those that do not. According to Sir William Crookes, the absorption of the X-rays by the colourless diamond and by the black form of carbon are alike.

Unfortunately, the experiment with these diamonds cannot be continued, as they were lost when my luggage was accidentally destroyed by fire when travelling through Belgium.

THE NATURE OF ALLOYS.†

THE research for which this Committee was formed is completed, and a summary of the results has been published in the *Proceedings of the Royal Society*, vol. lxi., p. 320. A copy of this paper has been sent to the Secretary of the British Association.

The work consists in a study of the chemical compounds and solid solutions to be found in alloys composed of copper and tin.

At least three series of solid solutions are formed during the solidification of these alloys.

* Read before the British Association (Section B), Belfast Meeting, 1902.

† Report of the Committee, consisting of Mr. F. H. Neville (Chairman and Secretary), Mr. C. T. Heycock, and Mr. E. H. Griffiths.

The first series, which may be called Alpha, consists of crystals, isomorphous with pure copper, and varying in composition from pure copper to an alloy containing about 9 per cent by weight of tin. These alloys solidify to a uniform mass, and apparently remain unchanged at all lower temperatures.

The second series, which may be called Beta, contains percentages of tin varying from 22.5 per cent to 32 per cent. Alloys between 9 per cent and 22.5 per cent of tin solidify as a complex of crystals of Alpha and of Beta. But all the alloys from 9 per cent to 32 per cent of tin undergo important re-crystallisations after they have wholly solidified, and their final condition below 500° C. is that of a complex of Alpha and a crystalline body which is probably Cu_3Sn .

Alloys from 32 per cent of tin to 57 per cent begin to solidify by the formation of a third type of crystalline solid solutions, which may be called Gamma. But the Gamma crystals break up at lower temperatures into a complex of crystals of the body Cu_3Sn and another substance. The alloy of the formula Cu_3Sn is apparently a solid solution when first solidified, and is not converted into the compound until a lower temperature is reached.

Gamma crystals containing more than 41 per cent of tin have the peculiarity that, in cooling, they break up into solid Cu_3Sn and a liquid. Between 57 per cent of tin and 93 per cent, the first solid that forms when the liquid alloy begins to solidify consists of crystals of Cu_3Sn ; but when the temperature falls to 400° C., these crystals become unstable, and a reaction takes place between them and the liquid, which results in their partial transformation into a body that is nearly, or quite, pure CuSn . Between 93 per cent and 99 per cent of tin the substance CuSn is the first body formed during solidification. Between 99 per cent and 100 per cent tin appears to crystallise first.

Assuming the alloys to have been cooled with sufficient slowness, we may summarise their condition at ordinary temperatures as follows:—

0 to 9 per Cent of Tin.—A uniform solid solution (Alpha) of copper containing tin, or, more probably, containing a compound, in solution.

9 to 25.5 per Cent of Tin.—A complex of large crystals of Alpha in a minute eutectic of Alpha and Cu_3Sn .

25.5 to 32 per Cent of Tin.—The same complex, but containing the Cu_3Sn in the larger crystals, and the Alpha only in the minute eutectic. At 32 per cent the alloy is pure Cu_3Sn .

32 to 38.5 per Cent of Tin.—A complex of Cu_3Sn and Cu_3Sn , or of two solid solutions of these substances. At 38.5 per cent the alloy is pure Cu_3Sn .

38.5 to 93 per Cent of Tin.—Large crystalline plates of Cu_3Sn coated with a body that is almost pure CuSn , the whole being immersed in a eutectic of this body and tin.

93 to 99 per Cent of Tin.—Large crystals of CuSn in a eutectic of this body and tin.

99 to 100 per Cent of Tin.—Large crystals of tin in the same eutectic.

STATISTICS CONCERNING THE TRAINING OF
CHEMISTS EMPLOYED IN ENGLISH
CHEMICAL INDUSTRIES.*

THE Committee decided that the best method of obtaining the desired statistics concerning the training of the chemists employed in English chemical industries was to send a circular letter, with a form for reply enclosed, to all those members of the Society of Chemical Industry

* Report of the Committee, consisting of Professor W. H. Perkin (Chairman), Professor H. E. Armstrong, Mr. G. T. Beilby, and Professor G. G. Henderson (Secretary).

who, so far as could be judged from the designations given in the list of members, occupy a position as manager or chemist in a works. This method was adopted because the great majority of the chemists engaged in technological work in this country are members of that Society, and because no other means of obtaining the information seemed practicable. The result of the inquiry was that more than half of those addressed sent replies to the circular. It is probable that a considerable proportion of those who did not reply are not engaged in chemical works, and therefore the accompanying statistics may be considered to give a fair idea of the present position.

Information concerning their course of training was received from 502 managers and chemists employed in English chemical industries. Of these, 107, or 21 per cent, are graduates, and 395 have not taken a degree; 111, or 22 per cent, are Fellows or Associates of the Institute of Chemistry.

The following figures give more detailed information:—

Number of graduates of a British university	59
Number of graduates of both a British and a foreign university	16
Number of graduates of a foreign university	32 (a)
	107
Number of non-graduates trained in a British university or university college ..	137 (b)
Number of non-graduates trained in a British technical college	165
Number of non-graduates trained in a foreign university or technical college ..	8
Number of non-graduates trained in evening classes, analysts' laboratories, works' laboratories, or otherwise .. .	85
	395
(a) Thirteen of whom studied also in a British university or technical college.	
(b) Twenty of whom studied also in a foreign university or technical college.	

The distribution of the chemists among the principal industries is shown in the accompanying table.

THE PROPOSED STANDARDISATION OF METHODS OF CHEMICAL ANALYSIS.*

By BERTRAM BLOUNT, F.I.C., F.C.S.

THERE has been evident of late years a tendency to apply the principle of standardisation to matters and in directions differing considerably from those in which the advisability of erecting and enforcing a standard is generally conceded.

The commonest examples of standardisation, namely, those of weights, of measures, and of money, have come into existence and are maintained by their manifest necessity. The standardisation of such things as gauges for wire and sheet, of screw threads, and the like, is also desirable. But there are limitations to the usefulness of standards, and these are reached when it is proposed to standardise sections of structural metals, sizes of test-pieces for mechanical tests, and methods of applying such tests. Whether these are to be standardised or not is largely a matter of convenience and expediency.

In mechanical testing it is recognised that the values obtained for strength in tension and compression, for elongation of a ductile test-piece, for resistance to shock and the like, are appreciably influenced by the conditions of the test, and in consequence there is something to be said in favour of standard methods of applying the tests. But there is no such justification for the adoption of standard methods in chemical analysis where the object in view is the determination in a given material of a definite substance; in such a case all methods which are chemically sound must give the same result.

But notwithstanding these obvious facts a desire has arisen in some quarters for the formulation of standard methods of chemical analysis and for their gradual imposition on the community of chemists; steps have been taken to secure this end, and it is necessary to decide whether this movement is laudable or not lest judgment should go by default.

The wish to establish standard methods of analysis is no new thing. From time to time propositions to this end have been put forward, but they have led to little result in this country. In America, however, they have been

* Abstract of a Paper read before the British Association (Section B), Belfast Meeting, 1902.

Industry.	Graduates—			Non-graduates, trained in—				Total.
	Of British university.	Of British and foreign university.	Of foreign university.	University or technical college.	Foreign university or technical college.	Evening classes, &c.		
Acids, alkalis, inorganic salts	9	3	5	20	19	2	20	78
Metallurgical (various)	1	—	4	19	13	—	14	51
Explosives	6	—	1	4	28	1	6	46
Dyeing and printing	3	—	—	13	16	—	5	37
Oils, fats, soaps, candles	2	1	3	11	9	1	5	32
Colours, pigments, oils, varnishes	8	1	2	6	5	—	6	28
Brewing and distilling	3	—	4	8	12	—	1	28
Fine chemicals, pharmaceuticals, confections	7	—	—	9	6	—	4	26
Sugar, starch, glucose	3	2	1	2	8	—	3	19
Cement, tiles, pottery	—	1	1	5	10	—	1	18
Aniline colours	2	3	7	2	2	1	—	17
Tar distilling	—	—	—	5	8	—	3	16
Paper, paper-pulp	—	—	2	3	3	—	—	8
Ghee, gelatin, size	—	1	—	4	2	—	—	7
Paraffin and paraffin-oil	—	—	—	3	4	—	—	7
Dyewood and tanning extracts	—	—	—	5	2	—	—	7
Cyanides and ferrocyanides	3	1	—	—	2	—	—	6
Glass	2	—	—	2	1	—	1	6
Coal-gas	—	1	—	—	3	—	2	6
Miscellaneous	10	2	2	16	12	3	14	59
Total	59	16	32	137	165	8	85	502

more favourably received, and standardisation of analytical methods for a number of materials has been advocated. Quite recently (in 1901), a committee of the New York Section of the Society of Chemical Industry approached the same subject in the same spirit, and has reported in favour of a standard method of analysis for cement and cement materials. I had the honour in May last of reading a paper before this body, in which I ventured to criticise the method in detail, and to dissent from the acceptance of a standard method.

It may be accepted that there is an increasing tendency to propose standardisation of analytical methods irrespective of the nature of the material to be analysed, and it remains to consider how this tendency has arisen and what effect it is likely to have if it continues to grow without opposition.

In the first place it will be seen that there are in this matter two schools of thought sharply differentiated. On the one hand, we have chemists so impressed with the necessity of avoiding discrepancies that they are anxious to set up standard methods stated in such detail that a faithful observance of the prescriptions by two workers cannot fail to secure identical results. And there are others less confident of the success of this system. This conflict of opinion arises in great measure from confusion of thought. There are many operations performed by the chemist which are from their nature arbitrary. To take an extreme case, it is evidently impossible to carry out the Mauméné test for oils, or to determine the flashing-point of petroleum, and to obtain concordant results without having recourse to a fixed procedure. In the examination of potable waters such arbitrary methods are in wide and general employment, and yield much useful information. Feeding-stuffs and manures afford a similar case. "Citrate-soluble phosphoric acid" is a meaningless term unless the conditions of its extraction are laid down. The fractional distillation of crude benzol and the return of its computed contents of benzene is another instance.

But this sort of codification, though useful and often necessary, has nothing to do with analysis. The object of the analyst is to determine with the best precision in his power the constituents of the substance which he is analysing. Sometimes he cannot do this, and is forced to have recourse to the determination of the properties of the substance; that is to say, he is compelled to apply to the material under examination various arbitrary tests. Standardisation for these tests is legitimate enough; standardisation of analysis implies that analysis is an arbitrary procedure not based on ultimate chemical facts; this view is too grotesque for discussion.

To turn to the much debated question of discrepancies in analytical results which is at the root of the whole matter.

In trade it is necessary frequently to appeal to the analyst, and if the results given by one analyst differ from those of another examining what is believed to be the same sample, much delay and expense may be incurred. The trader is usually unaware of the great difficulty which exists in procuring identical samples, and he is almost certainly ignorant of the slow and laborious character of all analytical processes having claim to be exact. From the former cause he is disposed to ascribe all differences to errors of the analyst, and from the latter he is inclined to urge greater rapidity of work than is compatible with accuracy. Sometimes from bad sampling or from hurried or unskilful work discordant or erroneous results may arise. Then the trader seeks a remedy, and believes that it may be found in a standard method of analysis. It is obvious that such a conclusion is fallacious. The same imperfection in sampling, the same hurried and unskilful work, will lead to the same erroneous results.

It is not likely that chemists who practice as independent consultants will submit to dictation even from a committee of their peers as to the methods they are to employ. A member of this branch of the profession asked to determine in some material a definite constituent is

either competent to choose a suitable method and to execute it accurately or he is not. If he is, a standard method is superfluous; if he is not, his early retirement is desirable and will probably occur from the operation of natural causes. But there are some consultants who are more or less identified with particular branches of the chemical trade—they are constantly called upon to carry out analyses or assays for the buyers and sellers of ores, metals, minerals, salts, and the like, and usually the results obtained by the buyer's and the seller's chemist are compared, and may be found to disagree.

Now here is a plausible case for the use of a standard method. It is argued that if the two chemists concerned were to use the same process, the likelihood of their disagreement would be diminished. There is even a limited truth in this; the likelihood of disagreement would be diminished to some extent because both operators would be exposed to identical chances of error instead of different chances. But the object of analysis is not to obtain a false concord by repeating a process, errors and all, with Chinese fidelity; it is to arrive at the truth as closely as knowledge and skill will allow, and one of the recognised methods of doing this is to vary the conditions of experiment.

A great part of the clamour for standardisation arises from the discrepancies which often occur between the results of different works chemists, and it appears that the "standard" methods, which have from time to time been proposed in the United States have the works chemist as their particular objective. These discrepancies are highly inconvenient, not only immediately to those concerned, but ultimately as tending to discredit the value of analytical control of manufacturing processes. When last in America I took some pains to ascertain the opinion of chemists on this point, and found that those who advocated the adoption of standard methods when confronted with the grave objections which can be urged against them, took refuge in the necessities of the works chemist, maintaining that only by the provision for him of standard methods could uniformity and accuracy be attained.

It cannot be denied that this view is defensible, but it is defensible on one ground alone, and that hardly likely to commend itself to the works chemist. His friends, in their anxiety to help him, infer that he is incompetent and needs to be guarded and guided like a schoolboy. Now, as a matter of fact, although often overburdened with work and most inadequately paid, the works chemist has done much to advance analytical chemistry, originating some methods and improving many others. It is neither just nor courteous to assert that he above all others is in need of direction and supervision from without, and that he is willing to barter his right of individual judgment according to his training and experience for a bundle of prescriptions. If, as is sometimes unfortunately the case, the works chemist is conscious of deficiencies, and welcomes "standard" methods, an argument is provided, not for the use of standard methods, but for the employment of better trained men. The fault is the manufacturer's, who hopes to obtain a chemist at the wages of a mechanic.

There are other results to be expected of wider and more general importance. Let us suppose that standard methods have been prepared and published with the consent, and under the direction of, some body of chemists of sufficient standing to make their pronouncements authoritative. The position of all chemists who ventured to disagree with the findings of this central body would become very difficult. In all cases of dispute, in all legal proceedings, these dissentients would be placed at a disadvantage; on them would be thrown the onus of proving, usually before a non-technical tribunal, that the method they preferred was as good as that of the central committee. If the dissentient were not able to convince the lay tribunal that his process was reliable a grave injustice might be done to him; if, on the other hand, he did so

succeed the standard process would be discredited. A whole vista of trouble and dispute can be foreseen. The endeavour here made to trace to its logical outcome the effect of standardisation is insufficient to show that such a scheme, even if it could be regarded as desirable, would lead to such complications as to render it impracticable.

But there are still further drawbacks of a kind yet more serious. If a standard process is adopted by a central authority it will be regarded by many as final, and all incentive to original inquiry in that direction will be removed. The desirability of a method radically different from that which is official will be regarded as a sort of chemical heresy, and will receive the treatment usually accorded to the heterodox. Now, analytical research, although not the highest form of chemical investigation, is of great value, both as a school of training and as a means of perfecting the chief instrument of the chemist, whose work, unless constantly controlled by its aid, would be little better than a maze of ingenious speculation. Anything calculated to discourage the criticism of existing analytical methods and the device of new ones is therefore much to be deprecated.

There is too good a case against the standardisation of methods of chemical analysis to make elaboration of argument necessary or wealth of instance and illustration desirable. The plain facts constitute so crushing an indictment, and appeal so strongly to every thinking chemist who is jealous of the dignity of his chosen profession, that their statement without ornament or rhetoric must suffice for conviction. But as there is nevertheless a respectable body of opinion in the contrary sense, it may not be superfluous to register a formal pronouncement that standardisation is undesirable except in cases where arbitrary methods of examination cannot be avoided.

Something may be done towards the construction of a positive policy. Revision of analytical methods is certainly desirable. This should be undertaken by committees of specialists acting under some central body, whose business it should be to examine and report on established and on new methods of analysis, each committee dealing with one of the great branches of technical analysis. The function of these committees would be critical and advisory, and no attempt would be made to erect a standard method. The object aimed at would be to ensure that the methods examined and finally approved should be at once reliable and practicable. In this way real analytical progress would be effected.

It will be seen that the programme suggested differs radically from that to which I have ventured to take exception. The latter involves the pronouncement of a defined method for the determination of a given substance; the former is directed to the discovery of latent errors and to the provision of processes free from these. In the one case, a set of rules is imposed from without; in the other, advance and improvement will take place from within, unfettered by canon and unhampered by rule. I think it not too much to say that the whole spirit of science, which is instinct with the love of freedom and the defiance of mere authority, will be found favourable to the plan which I have put forward, and will unconditionally condemn any attempt to standardise methods of chemical analysis.

Action of Hypochlorous Acid on the Metallic Chlorides.—W. van Tiesenholt.—The author describes several experiments which prove the production of free alkali in the decomposition of alkaline chlorides by hypochlorous acid according to the equation $KCl + ClOH = KOH + 2Cl$, which it can be seen is nothing but the inverse of the equation for the formation of hypochlorites. Experiments were also made on other chlorides, and especially the chlorides of the alkaline earths; the author draws some conclusions from his experiments, relative to the formula of chloride of lime and some of its reactions.—*Journ. f. Prakt. Chem.*, [2], vol. lxxiii., p. 39.

DETECTION AND APPROXIMATE ESTIMATION OF MINUTE QUANTITIES OF ARSENIC IN BEER, BREWING MATERIALS, AND FOOD-STUFFS.*

By WILLIAM THOMSON, F.I.C.

SINCE the arsenic in beer scare at the end of 1900 various methods, chiefly modifications of known processes, have been devised for the detection and estimation of minute quantities of arsenic in beer, malt, and other articles of food.

In London the Society of Chemical Industry formed a Committee for devising a standard or official method of testing for and estimating arsenic, whilst the Manchester section of the Society of Chemical Industry did the same thing.

It is impossible for a committee of this kind to work effectively. There is no sum of money placed at its disposal, and, as the devising of a standard method means the making of hundreds of experiments at a large expenditure of time and money, the members of such a committee, if they perform the duties of the office, will find the post no sinecure, and even if a process be formulated by such a committee it should not be suggested as final, but should be submitted to the most stringent criticism from chemists all over the world. The requirements of such a process are:—

1. That the results be obtained quickly.
2. That the personal equation should, as far as possible, be eliminated, so that two chemists would obtain as nearly as possible the same result when working on the same sample.
3. That as it yet remains uncertain as to how much arsenic may be taken without injury to health, cognisance should be taken of very minute quantities of arsenic up to, say, the $\frac{1}{10000}$ th of a grain of arsenic trioxide per lb. avoirdupois of solid or per gallon of liquid.

There are many methods by which the presence of arsenic may be detected:—

First.—The arsenic may be precipitated from beer or from water or acid solutions by sulphuretted hydrogen, and then treated by a variety of methods for its final determination. If the final precipitate be weighed, very large quantities of the material must be taken, and I think when an article contains the $\frac{1}{10000}$ th to the $\frac{1}{100000}$ th of a grain per lb. of solid or per gallon of liquid of the trioxide, that all will agree that the weighing method is not admissible, because these quantities are beyond the limits of direct gravimetric determination even when large quantities of the article are used for the analyses—and such are not usually available.

Second.—There are tests depending on volumetric determination, and in this we have a more accurate method than in the gravimetric. In this the arsenic liberated as arseniuretted hydrogen is passed into silver nitrate solution, and the precipitated silver determined volumetrically, or the arsenious acid left in the solution may be determined volumetrically, or, lastly, the metallic silver, which amounts to about six times the weight of the arsenic required for its precipitation, may be weighed and the weight of arsenic calculated therefrom, but when we are dealing with such minute quantities as the $\frac{1}{10000}$ th of a grain per gallon as a maximum, all these processes are inadmissible.

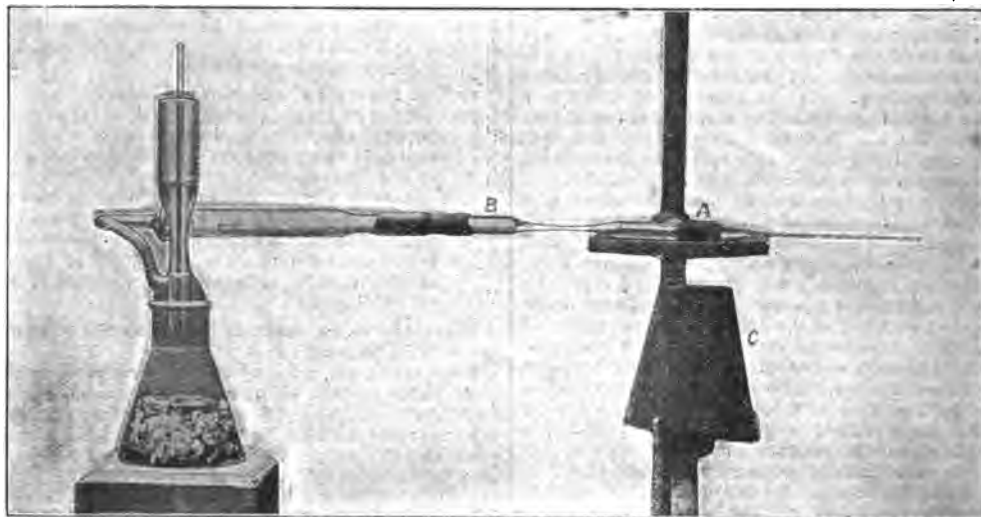
Third.—There are the tests depending upon the intensity of the colour obtained from deposits of arsenic as such, or of some chemical combination thereof, or of some other compound produced by the arsenic.

In this case we have the Gutzeit and the nitrate of silver tests.

* Read before the Manchester Section of the Society of Chemical Industry, May 2, 1902.

After experimenting for some months with these two processes I have thrown them aside as impossible, the extent of these deposits or colours being influenced by so many conditions. It appeared to me at one time that I had obtained in the Gutzeit test a good one for approximately determining minute quantities of arsenic. It was found necessary in order to obtain the fine canary-yellow colour of the mercury compound that oxygen should be present, and I suspended fine sewing threads through glass tubes of very narrow bore, and for different quantities of arsenic present I obtained different lengths of the thread coloured to about the same intensity, and although this seemed at first to give relatively good estimates of the quantities present, I soon found that the results were so irregular and different that I had to discard it; and after trying blotting-paper containing mercuric chloride over the top of the generating flask, this process I also discarded as inadmissible.

I arrived at the same conclusion when using silver nitrate on sewing threads and on paper in the dark. The results seemed more delicate than with the mercuric chloride in the Gutzeit test, but here again the results were so unsatisfactory and irregular that I also discarded this process.



I have made many experiments with Reinsch's test for the purpose of determining minute quantities of arsenic by depositing the arsenic on copper, and then heating the copper strips gently in small glass tubes, and comparing the densities of the white sublimates, but this process I found to be quite untrustworthy.

Experiments were made to measure the volume of the precipitate of mercury and of the metallic silver in tubes drawn out to a very narrow bore, but these also proved unsatisfactory, and the only one left which showed satisfactory results was the Marsh-Berzelius test. I have spent a great part of the last eighteen months in a research to discover the conditions required for giving the most reliable and accurate estimations of minute quantities of arsenic in malt, beer, and other articles of food by it.

A Joint Committee of the Society of Chemical Industry (London Branch) and of the Society of Public Analysts has already made a report with a view of formulating a standard method for detecting the presence of arsenic and approximately determining the quantity, and I propose to take the order of treatment which they have adopted for the purpose of criticism, because no process can be regarded as a standard till it has passed successfully through this ordeal. I appreciate fully the valuable work which some of the members of this committee have done,

especially in their attempt to found a standard process of analysis, and I propose as my contribution to the work to offer my criticism, differing from them where I must, and agreeing with them where I find it possible. I will take the order in which they present their report:—

Materials Required.

Hydrochloric Acid.—I agree that the acid sold as pure nearly always, if not always, contains arsenic.

The Joint Committee propose to purify that "pure" acid by diluting it to 1.10 specific gravity, then adding to it about 5 c.c. of bromine per litre of acid. Then sulphurous acid is added in excess; or hydrobromic and sulphurous acid may be added, and the mixture allowed to stand for twelve hours. This seems to me an extraordinary proceeding which requires explanation. What is the object of the bromine or hydrobromic acid? No satisfactory explanation is given. However, that procedure matters little so long as the required result can be obtained.

The acid is then boiled till about one-fifth has evaporated, and the residue can either be used direct or may be distilled, the whole of the arsenic having volatilised with the first portion of liquid which was boiled off.

I have carried out this procedure exactly as given, and after evaporating a fifth of the liquid I found that the arsenic had not been removed from the residue, and on continuing the distillation I found the further distillate to contain arsenic. This is unfortunate for the standard process devised by the committee.

My method of purifying hydrochloric acid is a very simple one. I do not require to use the purest acid which can be bought to start with. I simply dilute strong acid of any kind with about one-third its volume of water, although the dilution is not necessary. (If the acid be not diluted some water must be put in the receiver to condense or dissolve the HCl liberated as a gas at first). I put it in a retort, add to it about 1 gm. per litre of chromic acid, and distil. By preference I use a large stoppered glass flask placed on a sand-bath, a tube from the neck of which enters a Liebig's condenser, glass touching glass, and the distillate is collected. It is, however, saturated with chlorine.

I then pass a current of filtered air through the distillate, say about half a gallon, for about two hours by means of a water vacuum pump, when every trace of chlorine is removed, and no trace of arsenic will be found in the distillate.

On standing in most glass bottles hydrochloric acid

becomes faintly contaminated with arsenic, but the amount does not exceed the $\frac{1}{100}$ th of a grain per gallon dissolved during one month.

Sulphuric Acid.—The Joint Committee say sulphuric acid is more frequently obtained arsenic-free than hydrochloric acid. My experience has been very different from this, for I purchased sulphuric acid wherever I was told it was to be found pure all over the country, but I never obtained till very recently a sample of acid which was even approximately arsenic-free. Mr. Hart, of Messrs. Tennants and Co., of Manchester, sent me a sample which was practically free from arsenic, but it did give the shade of a mirror from 10 c.c., and it was therefore not absolutely free from arsenic.

The process given by the committee for purifying sulphuric acid is to take half a litre "pure for analysis," and add to it a few grammes of sodium chloride and distil from a non-tubulated glass retort, the first portion of 50 c.c. being rejected. This process I find also to give sulphuric acid free from arsenic, but the process is not a nice one to carry out. Hydrochloric acid vapours are copiously evolved, and if the acid contain any sulphurous acid, that objectionable body is not removed, and the result is that white sulphur mirrors are obtained by the use of the so-called purified acid.

The method I employ is, I think, much simpler than this, and deals with any sulphuric acid, whether "pure for analysis" or crude from the manufacturer.

Into a non-tubulated retort I put the sulphuric acid previously mixed with about a gramme to the litre of chromic acid or potassium permanganate. I distil and collect the distillate, which is free from any trace of arsenic or from sulphurous acid. If the tube of the retort has been contaminated with the crude acid during the process of filling, I throw away the first portion of the distillate. It is best to fill the retort with crude acid by means of a funnel and long glass tube, near the end of which is fixed a small section of bored cork to prevent the liquid at the end of the tube from touching the neck or tube of the retort when withdrawing the filling tube. The retort is set in a sand-bath standing on a ring burner, with an iron tray underneath, contained in a wooden box, and the neck of the retort passes through a hole in the end of the box; the top of the retort is covered with asbestos cloth, but not the tube. If the tube be covered all the way down it is liable to break suddenly from the end, the breakage running up towards the retort, and this produces an objectionable evolution of acid vapour into the laboratory. If the retort should break, the gas can be turned off from the outside, the lid placed quickly on the box, and a wet cloth thrown completely over it, which avoids any nuisance.

The whole of the distillate, even if the distillation be carried nearly to dryness, is free from any trace of arsenic.

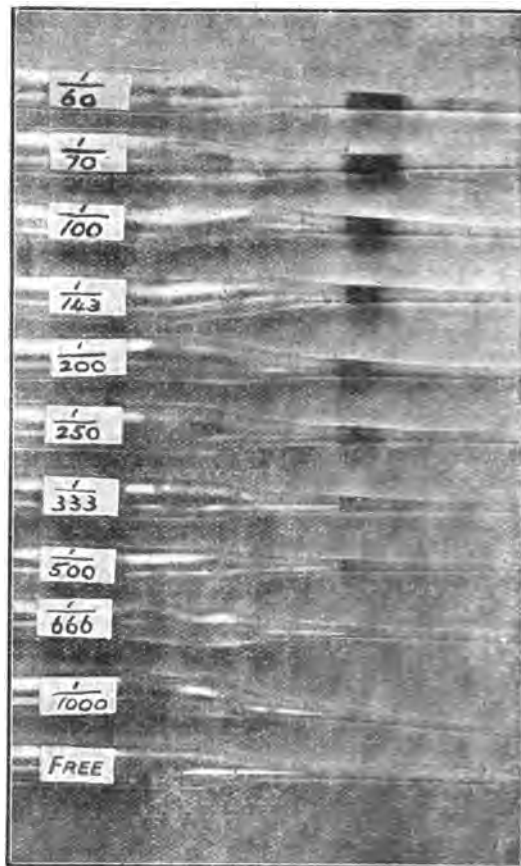
Nitric Acid.—The Joint Committee say this acid can, "as a rule," be obtained free from arsenic. I have never found any of the ordinary re-distilled nitric acid which I purchased from the dealers, except one, to contain any trace of arsenic, but it is advisable to make blank tests to prove that it is free. The one in question showed only the faintest trace.

Zinc.—The Joint Committee say that arsenic-free zinc is obtainable from chemical dealers. My experience has been that out of five or six samples sent by dealers as arsenic-free one is actually so. The zinc employed should be so pure that when placed in dilute sulphuric or hydrochloric acid very little hydrogen is liberated.

I find it necessary to add a little sulphate of copper to start and carry on the reaction. If the reaction commences at once vigorously the zinc contains some foreign metal, such as iron, in sufficient quantity to hold back considerable quantities of arsenic, so that although it may give hydrogen free from arseniuretted hydrogen it is yet quite unsuitable for the Marsh test.

It would be well if a reliable supply of zinc could be obtained free from iron or arsenic, and there seems no

reason why such should not be obtained electrolytically. Samples of electrolytic zinc which I have obtained yield no arsenic mirrors, but are contaminated with iron to such an extent as to be useless. If, however, this zinc be treated with acid, some pieces, especially those which have granulated in button form, produce rapid effervescence, and become black on the surface, from the formation of a deposit of metallic iron, whilst other parts, especially those which are most feathery, yield very little effervescence, and it is easy thus to pick out the good from the bad. The feathery pieces, being usually free from both arsenic and iron, are suitable for the Marsh test. I suggested to one manufacturer of electrolytic zinc that as the zinc was deposited on iron rollers the part furthest from the iron might be turned off on a lathe and sold to the pure-chemical manufacturers, but they did not see their way



to do this. Mr. Allen suggests that the zinc should be contaminated with a trace of iron to get it to work; this may be satisfactory, but the Committee do not explain how this contaminated zinc is to be prepared. Stirring the pure melted zinc with iron wire gives sufficient iron to it to render it useless for delicate arsenic tests, but the addition of one-and-a-half parts of metallic iron melted with 2000 parts of pure zinc might be employed. I have tried adding minute quantities of different metals to pure metallic zinc. Nickel answers the purpose best, as the smallest quantity of that metal produces the greatest effervescence, but on the whole I prefer the use of a solution of sulphate of copper, as the most constant mirrors are obtained by this method.

Chloride of Calcium.—The Committee suggest that this substance, used in the drying tube, often contains arsenic, which should be separated. They do not say that such

arsenic has been found to act injuriously on the test. I was told that molecular hydrogen was capable of removing arsenic from solutions, and tried the experiment, but my results showed that this was not the case.

Apparatus.—A 200 c.c. flask is recommended by the Committee in which to make the Marsh-Berzelius test. I have found that size of flask to be too large, and I use a flask of 50 c.c. capacity. The fitting of drying tube with cotton-wool and chloride of calcium I agree with, and also the use of the roll of dry blotting-paper containing acetate of lead. I prefer, however, to put this roll in the front end of the tube used for obtaining the mirror, and I do not see the use of the bulb at the end of the drawn-out portion, as shown in the Committee's diagram of this tube—but this is an unimportant detail. By my method I soften by the blow-pipe and draw out a piece of tube 200 m.m. long, $4\frac{1}{2}$ m.m. internal, and $6\frac{1}{2}$ m.m. external, diameter, from the middle, thus producing two tubes, each of 100 m.m. long, with 140 m.m. of drawn-out portion between them. The drawn-out portion is cut in the middle, leaving two tubes with 70 m.m. drawn out from each; the drawn-out portion for receiving the mirror being about 2 m.m. internal diameter.

At 70 m.m. from the drawn-out end the glass is again softened and drawn out for 30 m.m., and in the remaining 30 m.m. portion of the tube at the end is inserted a roll of filter-paper previously soaked in a strong solution of lead acetate and dried. I do not regard the passage of the gas through a solution of acetate of lead as satisfactory, as some of the arseniuretted hydrogen is dissolved and retained by the solution.

When the test has been completed the point of the drawn-out portion is sealed, and the drawn-out portion at the other end is also sealed by the blow-pipe. I have examined mirrors which have been thus sealed in hydrogen in comparison with others left unsealed, and have not been able to observe any difference between them after many months. But I am not prepared to say that the mirrors may not fade by exposure to light and air, and under any condition I think it is desirable to seal up the tube and preserve the mirrors in an atmosphere of hydrogen.

I think the arrangement devised by me for introducing the solution to be tested is an improvement on the arrangement with the glass tap suggested by the Joint Committee. In this I grind the end of a glass rod into the end of the thistle tube entering the Marsh-Berzelius flask, which has previously been rounded in slightly, by softening the end in a Bunsen flame; on raising the rod the liquid flows in, and on letting it down the flow stops. By this means no column of liquid is left in the tube, as is the case when a glass tap is used, and no gas can rise through this simple stopper.

The Joint Committee think rather denser mirrors are obtained with minute quantities of arsenic when using hydrochloric acid than when using sulphuric. I have devoted some attention to this, but have not been able to get such results. I think it is possible that hydrochloric acid, which cannot usually be obtained quite free from arsenic by the Joint Committee's method, may account for this difference. At all events I have not been able to find any appreciable difference in the mirrors obtained for the same quantity of arsenic when working with hydrochloric and with sulphuric acids respectively.

The Joint Committee's suggestions as to testing beer, yeast, &c., directly and without destroying the organic matters do not seem satisfactory to me. No mirror is shown unless the article contain comparatively large quantities of arsenic, and this can only be regarded as a very rough test.

My experiments show that the direct testing of beers containing less than $\frac{1}{10}$ th of a grain of arsenic trioxide per gallon is utterly untrustworthy. In some cases beers containing that quantity give a slight yellow mirror, and in some cases with $\frac{1}{10}$ th they give no mirror, whilst this amount is easily detected and estimated after the organic matter is destroyed.

Destruction of the Organic Matter.—The old process suggested by the Joint Committee for this purpose seems to me clumsy, and involves more work and attention than the one used by me. The Joint Committee's process involves treatment by nitric acid and afterwards charring the organic matter with sulphuric acid, the charred mass being then washed with water and the washings concentrated. Tests carried out with this process in comparison with my own, in which all the organic matter is removed by oxidation, showed that the former process takes a longer time and gives considerably smaller arsenic mirrors than the latter. I found the direct electrolytic method, although generating arseniuretted hydrogen when the arsenic is present in considerable quantities, was quite useless for detecting minute quantities of arsenic.

I understand that the Joint Committee's processes were tested by other members of the Committee on samples containing known quantities of arsenic. It is unfortunate that the results obtained by the different members of the Committee have not been published.

The following is the process which I have found to be most satisfactory for approximately determining small quantities of arsenic in beer, malt, &c.

Process for Estimation of Arsenic in Beer and Stout, Malt, Caramel, &c.

Beer.—Take of the sample 50 c.c. and evaporate to a syrup on a sand-bath or iron plate in a 200 c.c. Jena glass flask. Add 25 c.c. strong nitric acid and 5 c.c. strong sulphuric acid; place on a hot sand-bath, having taken away the flame; allow the first violent action to subside, the acid fumes from which may be drawn away through a glass tube in the mouth of the flask by a water Sprengel pump through a solution of caustic soda in a Wolff's bottle. Then apply a Bunsen flame to the sand-bath, and evaporate till the liquid begins to darken. When this occurs add strong nitric acid in quantities of 3 c.c. at a time (the total quantity of nitric acid required varies from 30 to 50 c.c., depending on the quantities of organic matter present), until on further heating it continues colourless, and fumes strongly of sulphuric acid; cool, dilute with 10 c.c. of water, and boil down to break up the nitro-sulphuric acid formed. When cold, dilute with 10 c.c. of water, and deliver into the Marsh-Berzelius apparatus, the capacity of which should not exceed 50 c.c. The gas evolved should be dried over calcium chloride contained in one tube 70 m.m. long by 8 m.m. diameter.

Testing Reagents.—A blank on the reagents and apparatus used should be made by boiling down 100 c.c. HNO_3 and 5 c.c. H_2SO_4 till all nitric is expelled, diluting and boiling down (this procedure removes every trace of nitric or nitrous acid), again diluting, and testing in the Marsh-Berzelius apparatus as above described.

Process for Malt, Sugar, Caramel, Hops, Yeast, &c.—Take 5 grms. of malt or other solid organic substance, add 25 c.c. HNO_3 , and heat gently till the first violent action is over; then add 5 c.c. sulphuric acid, and proceed as for beer (a total of from 50 to 75 c.c. of nitric acid will be required).

The Marsh-Berzelius Apparatus (50 c.c. flask) should contain about 20–25 grms. zinc. The CaCl_2 in the drying tube should be renewed as soon as the first few pieces become wet.

Action is started by adding 5 c.c. of dilute sulphuric acid (10 parts concentrated sulphuric, 20 of water, and 1 part of a 10 per cent solution of pure crystallised copper sulphate). Allow the evolution of gas to go on (the exit tube for the hydrogen being heated in the usual manner by means of a small Bunsen flame) until the hydrogen nearly ceases to be evolved (I see no advantage in removing the air from the apparatus by means of hydrogen generated in another apparatus); then fill up the tube and funnel of the Marsh-Berzelius apparatus with the solution previously treated as above, and allow the whole to run

in if only a very minute quantity of arsenic is supposed to be present; or run in an aliquot part if a larger quantity is supposed to exist. In about fifteen minutes the flask is washed with 5 c.c. more of the above acid, which is added to the hydrogen flask in small quantities at a time to keep up the evolution of gas for a total of from thirty to thirty-five minutes after the first introduction of the previously treated beer, by which time, with this sized apparatus, all the arsenic will have passed off.

The hydrogen flame at the end of the drawn-out portion should be about 2 m.m. long, and maintained as constant as possible; too slow a flow almost invariably gives double mirrors, too fast a flow gives irregular ones difficult to compare with the standards.

Zinc.—Twenty grms. of zinc should be tested by the action of 30 c.c. of dilute H_2SO_4 —one of acid to two of water—containing a little copper sulphate to start the reaction as above described; there should be absolutely no trace of arsenic mirror on the drawn-out portion of the glass tube.

Another experiment should then be made, adding a minute quantity of arsenic, say equal to 1/500th of a grain per gallon (when working on 50 c.c.), equal to 0.0029 part per 1,000,000, or an actual weight of 0.00143 m.grm., and compared with a standard tube to make sure that the zinc contains nothing which will hold back minute quantities of arsenic.

Glass tube should be about 4 1/2 m.m. internal diameter, and about 6 1/2 m.m. external. It should be drawn out in the middle and at the end, the length of the drawn-out portions respectively being 30 m.m. and 70 m.m., and the portion of tube between the drawn-out portion being 60 m.m. The diameter at the beginning of the drawn-out portion for receiving the mirror is about 2 m.m. internal diameter.

A piece of fine iron gauze 20 m.m. wide is wrapped round the tube at the point A shown in the figure, and heated by a Bunsen flame. This is a more satisfactory plan than applying the flame directly to the tube, and conduces to the formation of more uniform mirrors.

A roll of lead acetate paper is introduced to absorb any traces of sulphuretted hydrogen which may be formed.

Bunsen Flame.—This should be about 4 in. long, and protected till near the point by a conical iron or copper chimney of about 2 1/2 in. high by 2 1/2 in. at the lower, and 1 1/2 in. diameter at the higher part of the cone C.

Heating the Tube.—This should be heated from the shoulder, or drawn-out portion, for about 1/2 in. at the point A.

Standard Mirrors for comparison are made by introducing into the apparatus known quantities of arsenic, say, commencing with 1/50th of a grain per gallon when working on 50 c.c. A convenient method of preparing the standard mirrors is to employ a solution of arsenious acid containing 0.007145 of a m.grm. of As_2O_3 per 1 c.c. One c.c. of this solution is equal to 1/100th of a grain of As_2O_3 per gallon when using 50 c.c., or 1/100th of a grain per lb. when using 5 grms. of material. This solution is suitable for standards between 1/50th and 1/100th of a grain per gallon, and a solution of one-tenth this strength may be employed for the standard mirrors between 1/100th and 1/1000th of a grain per gallon.

The mirrors I have found most useful are 1/50th, 1/66th, 1/88th, and 1/100th of a grain per gallon, corresponding to 2.0, 1.5, 1.25, and 1.0 c.c. of the stronger solution, and 1/111th, 1/125th, 1/143rd, 1/166th, 1/200th, 1/250th, 1/333rd, 1/500th, 1/666th, and 1/1000th of a grain per gallon, corresponding to 9.0, 8.0, 7.0, 6.0, 5.0, 4.0, 3.0, 2.0, 1.5, and 1.0 c.c. respectively of the weaker solution.

The second figure gives photographic representations of different amounts of arsenic deposits from beer. The figures on the tubes show fractions of a grain per gallon, using 50 c.c. of beer in each test.

Cacodylic Acid.

I have made experiments with a view of finding whether

the arsenic contained in this compound could be estimated by my process:—

1. I added directly cacodylic acid representing 1/100th of a grain of As_2O_3 to the Marsh-Berzelius apparatus. This would have given a very heavy mirror if the arsenic had been eliminated as arseniuretted hydrogen; but an arsenic mirror was formed equivalent to about one-tenth of the quantity taken, which was probably not due to the arsenic in the cacodylic acid.
2. Cacodylic acid, equivalent to 1/1000th of a grain of As_2O_3 was digested with nitric and sulphuric acids in the ordinary way, and also with fuming nitric acid, heated under pressure in a hermetically sealed glass tube, the nitric acid being then removed in each case, and the solution "marshed," but no arsenic from the cacodylic acid was indicated in either case.
3. Fifty c.c. of the solution containing cacodylic acid equivalent to 1/100th of a grain of As_2O_3 per gallon were treated with nitric and sulphuric acids in the ordinary way, along with potassium permanganate (which was suggested by Villiers for breaking up organic compounds), by using 20 c.c. of a half per cent solution of potassium permanganate in addition to the sulphuric and nitric acids; the cacodylic acid was thus destroyed, and its equivalent arsenic mirror was obtained.

NOTE ON THE ESTIMATION OF SODIUM CHLORIDE IN SOAPS.

By A. R. WARNES.

SOME discordant results having been obtained when examining a bar of soap for sodium chloride, the following experiment was carried out to prove the accuracy of the method used:—

A sample of soap was made in the laboratory, using chlorine-free materials. Ten grms. of this pure soap were taken and dissolved in water in a 6-oz. beaker. To the solution a known quantity of pure sodium chloride was added, the fatty acids then thrown up with sulphuric acid, and the beaker and contents heated on the water-bath until the aqueous layer became clear. The beaker was allowed to cool, then the cake of fatty acids was pierced in two places and the aqueous layer poured into a separating funnel. The fatty acids and the interior of the beaker were then well washed with cold water, and the washings added to the liquor in the funnel. Petroleum ethers (all over under 80° C.) were added, and the funnel well shaken in order to extract traces of dissolved fat acid. After separation, the aqueous layer was run into an 8-oz. conical flask, one drop of phenolphthalein indicator added, and ammonium hydrate added until solution became just pink. The flask was then placed on the water-bath and left until any retained petroleum ether was driven off, and then boiled until the vapours showed no reaction with red litmus-paper. The liquor was then cooled and titrated in the usual way with N/10 $AgNO_3$. Afterwards a little nitric acid and excess of silver nitrate were added, and the sodium chloride determined gravimetrically.

The results of two experiments are tabulated below.

	NaCl added. Grm.	NaCl found volumetrically. Grm.	NaCl found gravimetrically. Grm.
1.	0.0423	0.0427	0.0422
2.	0.030	0.0307	—

Differences of Potential Contact.—Pierre Boley.—The author examines the action of piles formed of amalgams of the metals ordinarily used with sulphuric acid. He finds that the differences of potential between the amalgams is so small as to be only a few millivolts—values which are of the same order as the possible errors of the experiment.—*Comptes Rendus*, cxxxv., No. 11.

PROCEEDINGS OF SOCIETIES.

ROYAL SOCIETY OF NEW SOUTH WALES.

THE General Monthly Meeting of the Society was held at the Society's House, No. 5, Elizabeth Street North, on Wednesday evening, July 2, 1902; Prof. WARREN, M.Inst.C.E., Wh.Sc., President, in the Chair.

The following paper was read:—

"Notes on Two Chemical Constituents from the *Eucalypts*." By HENRY G. SMITH, F.C.S., Assistant Curator, Technological Museum.

In this paper the author records the results of continued investigations on the ester (geranyl-acetate) contained in the oil of *Eucalyptus Macarthurii*, and also on the oil itself. This data shows that the ester does not fall, at any time of the year, below 60 per cent, and that the amount of free alcohol, considered as geraniol, diminishes in amount as the ester increases. The greatest amount of naturally formed ester occurring at any time of the year was 74.9 per cent in September, but the free alcohol was only 6 per cent at that time. It has been found from numerous determinations that when the oil is acetylated the ester content will be but little removed from 80 per cent. The oil does not contain phellandrene at any time of the year, and eucalyptol appears to be always absent. Eudesmol is always present, but as it varies in amount, the specific gravity of the oil varies also. The crude oil appears to be always slightly dextro-rotatory. From the results of investigation of the oil obtained from over 200 distinct species of *Eucalypts*, this is the only one found to contain this valuable oil. The author also shows that the original formula for the quercetin glucoside, myrticolorin ($C_{27}H_{48}O_{16}$), obtained from the leaves of *Eucalyptus macrorhyncha* was correct (*Trans. Chem. Soc.*, 1898, p. 697). This formula has been confirmed by Mr. A. G. Perkin, who has shown (*Trans. Chem. Soc.*, 1902, p. 477) that his own osyritrin, and also Mandelin's violaquercitrin have the same formula, and are identical substances with myrticolorin. They all form quercetin and glucose on hydrolysis.

The following is an abstract of the first Science Lecture of the present Session, delivered on June 30 by F. TIDSWELL, M.B., M.Ch., D.P.H., Health Department, on "The rôle of Bacteria in the Production of Disease."

The lecture opened with an account of the discovery of bacteria two centuries ago by the Dutchman Anton van Leeuwenhoek. Allusion was then made to the subsequent controversies concerning the origin of bacteria, and their influence in causing disease, and the manner in which their final settlement was effected by the researches of Louis Pasteur some thirty years ago. Attention was then directed to Pasteur's demonstration of the causal micro-organism of a disease affecting silkworms, and of Davaine's observations on the microbe of anthrax. It was shown how these results stimulated investigation of the germ theory, and how the proof of its correctness was rendered possible by the elaboration of exact bacteriological methods by Robert Koch. Mention was then made of the species of bacteria which produce disease in man. There followed a brief account of the structure and functions of bacteria in general, and the specialisation of some forms into producers of disease. The consideration of the occurrence of these bacteria in the surroundings of the sick, and even upon the healthy skin, led up to an account of Lister's institution of methods for preventing their entry into wounds, and its development into the modern system of aseptic surgery and of the principles underlying the hospital isolation of the infectious sick, and disinfection. The means by which infectious bacteria gain access to the surface of the body, and the events connected with their entrance to, and development in, the internal tissues were dealt with at length. It was pointed out that the manifestations of these diseases depend upon the injury done to the tissues by the poisons or toxins produced by the

bacteria, and that recovery from them is consequent on the elaboration of neutralising substances or antitoxins by the cells of the body. Reference was then made to the after-effects of these diseases, including the immunity against subsequent attacks, and in this connection attention was called to the use of viruses, vaccines, any prophylactics as preventives, and of antitoxic serums as cures for infectious diseases.

OBITUARY.

JOHN HALL GLADSTONE, Ph.D., F.R.S.

It is with great regret that we announce the death of Dr. John Hall Gladstone, which has just occurred suddenly and unexpectedly on Monday morning last, the 9th inst.

Dr. Gladstone was born on March 7th, 1827, and was therefore in his seventy-sixth year. He first studied chemistry at University College, London, under Prof. Graham, then at Gießen under Prof. Liebig. He took the degree of Ph.D. in 1848, and was elected a Fellow of the Royal Society in 1853; he was the first President of the Physical Society on its foundation in 1874, and was President of the Chemical Society from 1877 to 1879.

Dr. Gladstone's first paper was published in the year 1845; it was entitled "Contributions to the Chemical History of Gun-cotton and Xyloidine." Since that time he has been constantly engaged in scientific research, principally in chemistry and optics, and the points of contact between these two sciences. In conjunction with the late Dr. Alfred Tribe, he published a large number of papers on chemical activity with regard to electrolysis, the copper-zinc couple, &c.

Dr. Gladstone may be said to have died "in harness," as his last paper, "On Fluorescent and Phosphorescent Diamonds," and one in conjunction with Mr. W. Hibbert, "Colloids of Zirconium, compared with those of other Metals of the Fourth Group," were only quite recently read at the meeting of the British Association at Belfast, and appear in this number of the CHEMICAL NEWS.

But it was not only in scientific circles that Dr. Gladstone was well known; for many years he had been engaged in various philanthropic and religious movements, and from 1873 until 1894 he represented Chelsea on the London School Board, of which he was also Vice-Chairman.

Amongst other books of which he was the author, we may mention "The Biography of Michael Faraday," "Spelling Reform from an Educational Point of View," and the "Chemistry of Secondary Batteries."

In private life his pleasant face and genial manners have endeared him to a large circle of friends. He was also a well-known and constant attendant at many scientific meetings, where his kindly presence will be greatly missed.

PROFESSOR JOHN JAMES HUMMEL, F.I.C.

In the death of Professor Hummel, F.I.C., the dyeing world has lost one of its most brilliant exponents. He was the pioneer teacher of this subject, an arduous worker, and scientific investigator. The experience acquired in the works as manager, and also as practical chemist, combined with his training in the Technical School at Zürich under Stadeler and Wislicenus, qualified him for the work of initiation in the Dyeing Department of the Yorkshire College, with which his name will always be associated.

John James Hummel was of Lancashire birth, being born in Clitheroe, but his father was a native of Switzerland, and a technical chemist, occupying positions of responsibility in Dyeing and Printing works at Elbeuf, Paris,

and Rouen, and subsequently chemist in the printing works of James Thomson, F.R.S., of Clitheroe. The influence of Mr. Thomson brought him into touch with many eminent scientific men, among them being Frederick Steiner, John Mercer, Walter Crum, Daniel and Camille Koechlin. It was only natural that Mr. Hummel should inherit ability for scientific pursuits, and that, though for a time he entered business, he should manifest decided taste for some branch of technical chemistry.

After his training under Mr. G. Knecht, B.Sc., and a short term in a Manchester office, he was three years in the Polytechnic School of Zürich, where he obtained his diploma, and adopted the profession of his father, entering a calico print works as chemical adviser. Dr. Grace Calvert, F.R.S., offered him an assistantship in the Manchester Royal Institution Laboratory, but this he declined, accepting the post of chemist in the print works of Messrs. James Black and Co., near Glasgow. His faculty for teaching, and interest in, technical education were first exercised whilst with this firm, for he formed a class in chemical technology, bearing specially upon dyeing and printing, imparting instruction gratuitously, and not a few of the young men attending attribute their success in life to this training. Later, he was manager in a print works in Lancashire, and for a firm of woollen-yarn dyers in Scotland.

In 1879, the Clothworkers' Company of the City of London extended their departments at the Yorkshire College by establishing a school of dyeing, and Mr. Hummel, early in 1880, was appointed the head of the new department. His success as a teacher was soon recognised, and in 1885 well-equipped buildings with suitable lecture-room, museum, experimental dye-house, and laboratories were erected. Since that time, students have been trained under Professor Hummel from all parts of the United Kingdom, the Continent, America, Canada, Japan, and other countries.

Some four years ago Professor Hummel advocated the importance of scientific research in the chemistry of dyeing, and as a result the research laboratory and other extensions of the dyeing department were equipped and formally opened in May, 1900. These also comprise pattern and practical dye-houses, so that the equipment which Professor Hummel designed and carried out with a mastery of detail is such as to be superior to that of any other school of dyeing in the world, and to effect the teaching of dyeing in the fullest degree, both in its pure scientific and practical phases.

The commencement of this work, and of the woollen and worsted yarn-spinning branches of the textile industries department, have produced an educational unity between the Clothworkers' departments which has made it feasible to teach the science and practice of the manufacturing of all kinds of woollen, worsted, and mixed fabrics, not yet possible to the same extent in any Continental or American school. The experiments conducted are of such a nature as to show the influence of the dyeing processes upon the nature of the material, the spun yarn, and the woven fabric. Many fields of research are, between the departments, being investigated. The able manner in which the dyeing extensions have been designed, and the work sub-divided into pure scientific research, experimental dyeing, and pattern and practical dyeing, have made the course of study increasingly valuable. So far as the practical dyeing of materials, yarns, and cloth was concerned, the desire of Professor Hummel was to perform it with the same method and business accuracy as characteristic of actual works.

As a lecturer and teacher of experimental science the late Professor possessed natural and trained ability. Fluent and clear, and competent of expounding every detail of the subject dealt with, his style was methodical, analytical, and concise. In his lectures he had recourse to his experience in the works, and, recognising the value of illustration and experiment, he made ample use of diagrams, lantern-slides, and specimens. Those who

have studied under him have many incidents by which to recall his kindness, willingness to help in a difficulty, and to render to the earnest seekers after knowledge the best of all assistance, sympathetic encouragement.

Mr. Hummel has made several valuable contributions to literature on the science and technology of dyeing. His principal work, "The Dyeing of Textile Fabrics," has been translated into German, Italian, and Japanese, and his articles on dyeing in the "Encyclopædia Britannica" and in various scientific and technical journals, are well known to the dyeing industry. In recent years his energies had been so absorbed with the new buildings that his literary activities had to be curtailed, but I have reason to believe that he was gleaning information and making experimental research which would have ultimately been published.

None who knew Mr. Hummel intimately can readily forget his uniform thoughtfulness, true sympathy, and friendship. He was in every sense devoted to the task with which his life was so fully associated, but this did not prevent him manifesting enthusiasm in the solution of the difficulties of others. He was highly esteemed by his colleagues, by the Textile and Dyeing Committees, and the Clothworkers' Company. A sincere and true man, he was faithful to duty, and lived for the high purpose of extending scientific knowledge and for the exercise of influences which would better humanity. In the dyeing department of the Yorkshire College, which his endeavour has organised, there is a memorial of industry and of lasting service rendered to the science and art of dyeing.

NOTICES OF BOOKS.

Thirty-eighth Annual Report on Alkali, &c., Works. By the CHIEF INSPECTOR. London: Eyre and Spottiswoode.

THIS report, presented to the Local Government Board under the Alkali, &c., Works Regulation Act, summarises the state of chemical industry in the United Kingdom during the year 1901. Compared with 1899 and 1900, the Inspector has to record a reduction on the whole, both in the number of works and of processes. The report includes a valuable paper by Dr. Affleck on "The Composition of Gases Evolved in the Manufacture of Superphosphate Manures, and a more Accurate Method of Determining their SO_3 Equivalents," which will be read with much interest by all chemists; and also the results of further experiments, carried out in the Chief Inspector's own laboratory, on the action of ferrous thiosulphate on sulphuretted hydrogen; the methods employed being closely allied to those described in last year's report.

Wireless Telegraphy. By G. W. DE TUNZELMANN, B.Sc. Third Edition. London: The Office of Knowledge, 1902.

THIS "Popular Exposition" is based upon a series of papers on wireless telegraphy which appeared in *Knowledge* during the year 1900, but a good deal of matter has been added to bring them up to date. The book is intended primarily for the general reader who is practically ignorant of physics. Regarding it from this point of view, we are inclined to think that such a reader might well have been supposed to possess more knowledge of the elements of mathematics, though perhaps it would be difficult to set a limit to the introduction of formulae if once begun. There is sometimes a great want of cohesion in the treatment of the subject, and some paragraphs will undoubtedly have to be re-read to discover the author's meaning. Many of the illustrations are borrowed from Lodge's "Modern Views of Electricity" and the *Electrical Review*.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 11, September 15, 1902.

Electric Resistance of Badly-conducting Bodies at very Low Temperatures.—Edmond van Aubel.—The electric resistance of metals and alloys at very low temperatures has been measured by Dewar and Fleming and by Arsonval. It is found to diminish considerably in proportion as absolute zero is approached. The author now examines the electric resistance of badly-conducting bodies, such as certain oxides and sulphides, whose electric conductivity increases during the elevation of temperature from 0° to 100°. The first experiments were carried out on a very homogeneous specimen of pyrites, FeS₂, and it was found that the resistance of the pyrites increased as the temperature was reduced, but it still conducts to a certain extent even, in liquid air. The author is now continuing his researches on other sulphides.

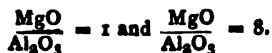
Formation of Liquid Drops and Tate's Law.—Ph. A. Guye and F. Louis Perrot.—The authors find that, under ordinary experimental conditions, the weight of drops issuing from the same orifice and rapidly formed is greater than that of those formed slowly; also, if the duration of formation increases, the weight of the drop tends towards a limit, which practically does not vary if the time of formation is long enough. With a tube of 3.17 m.m. of exterior diameter, the weight of the drop is independent of the time of formation only if this is thirty to forty seconds, or, for certain liquids, even from twenty to twenty-five seconds.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxvii., No. 5.

Ammonio-calcic Phosphate.—Henri Lasne.—Already inserted in full.

Action of Oxide of Bismuth on various Metallic Solutions.—J. Aloy.—On boiling recently precipitated oxide of bismuth for one hour with saline solutions, the author has observed that with ferric salts the Fe is only completely displaced in weak solutions. With ferrous salts the Bi displaces the Fe from the chloride, even in the cold, but much more easily when boiled; it forms a greenish white precipitate, easily oxidised, consisting of a mixture of basic salts, or perhaps of a mixed basic salt; the amorphous form of the precipitate renders it impossible to decide between these two hypotheses. As with iron, aluminium is only completely displaced in very dilute solutions. With manganese the displacement is partial. Copper is partially displaced from its concentrated solutions of the chloride, nitrate, and sulphate, in the form of greenish blue basic salts; in the case of the acetate the black hydrate is precipitated. The metals cobalt, nickel, lead, zinc, and cadmium also are partially displaced from their solutions. The anhydrous oxide produces the same reactions, but much less readily than the precipitated oxide.

An Electric Heating Apparatus.—M. Guntz.—Instead of using a fine platinum wire embedded in asbestos or enamel for the purpose of obtaining high temperatures of 1200° to 1300°, the author used a brasque of aluminate of magnesia or lime for covering the wire, and found that it protected the wire from all alteration. This method has the great advantage that apparatus of any form can be heated; muffles and furnaces of any desired shape can be constructed very quickly, and if desired the heat can be distributed unequally by increasing or decreasing the spaces between the spirals, or the section of the wires. The brasque of aluminate of magnesia was made of two mixtures:—



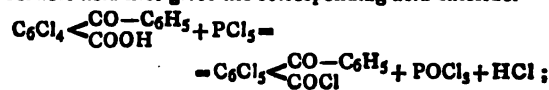
The calcined alumina and magnesia are finely pulverised and passed through a silk sieve. The paste is made with a little water and gelatinous alumina, which causes it to be slightly plastic; a dilute solution of acetate of alumina can also be used. The brasque of aluminate of lime is easier to make; it is formed by mixing 1 part of strongly calcined Al₂O₃, 1 part of Al₂O₃ produced by the incomplete calcination of ammonia-alum, and 1 part of aluminate of lime obtained by heating equal weights of pure lime and alumina to redness. Care must be taken that no sensible amount of silica is present.

No. 6.

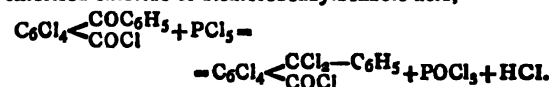
Action of Hydrates of Copper on Aqueous Solutions of Metallic Salts.—A. Mailhe.—Already noticed.

Action of Binoxide of Sodium on the Metals of the Platinum Group.—MM. Leidie and Quennessen.—Already noticed.

Action of Pentachloride of Phosphorus on Tetrachloro-*o*-benzoylbenzoic Acid.—L. Tétry.—The action of pentachloride of phosphorus on tetrachloro-*o*-benzoylbenzoic acid first gives the corresponding acid chloride:—



but the pentachloride of phosphorus, under the conditions of the experiment, reacts on the latter, giving the tetrachlorised chloride of dichlorobenzoylbenzoic acid,—



Direct Analysis of Essence of Pennyroyal.—L. Tétry.—Essence of pennyroyal was submitted to fractional distillation *in vacuo* under a pressure of 20 m.m., and three portions were obtained; the principal portion, boiling at 110°–112°, consisted of the raw pulegone. The author describes two methods for the examination of this product. The first consists of transforming the menthol into benzoate, easily separated from the pulegone by distillation *in vacuo*. The second method consists of transforming the pulegone, and the ketones which may accompany it, into oximes, of which the boiling-points are naturally much higher. These oximes are separated by distillation *in vacuo*, and the menthol looked for in the most volatile portion. The other portions of the essence of pennyroyal were submitted to fractional distillation *in vacuo*; a considerable quantity was obtained boiling at 60°–90° under 20 m.m., and another passing over at 90°–110° at the same pressure.

Action of the Alcoylcyanacetic Ethers on the Diazo Chlorides.—M. Favrel.—Already noticed.

Action of the Acidlycyanacetic Ethers on the Diazo and Tetrazoic Chlorides.—M. Favrel.—Already noticed.

Chemical Modifications in Plants submitted to the influence of Chloride of Sodium.—E. Charabot and A. Hébert.—Already noticed.

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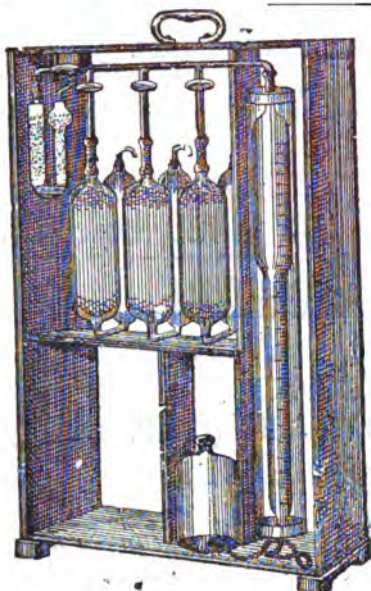
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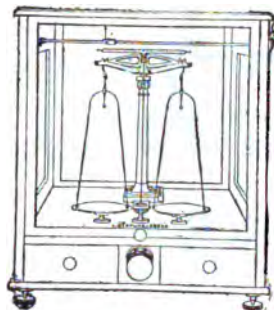
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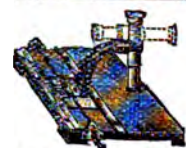
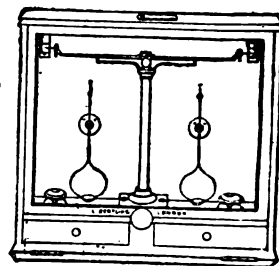


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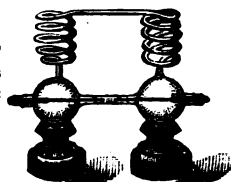
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THE CHEMICAL NEWS.

VOL. LXXXVI., No. 238.

THE TELLURIC DISTRIBUTION OF THE ELEMENTS IN RELATION TO THEIR ATOMIC WEIGHTS.*

By WILLIAM ACKROYD, F.I.C., F.C.S., &c.,
Public Analyst for Halifax.

ALL that is known of the comparative abundance of the elements in the earth may be said to be comprised in such tables as are given in Sir H. E. Roscoe's "Elementary Chemistry," p. 8 (based on Bunsen's analyses of the volcanic rocks of Iceland), and in *Bulletin* 148 of the U.S. Geological Survey, 1897, p. 13. The former gives the percentage by weight of the eight elements which are most plentiful in the primary rocks, while the latter similarly deals with twenty-one elements, but the question of distribution in relation to atomic weight is not dealt with.

From a critical survey of Prof. F. W. Clarke's figures, I find some indication that the elements of low atomic mass exist in larger proportion than those of higher mass; the latter, indeed, on account of scarcity, often finding no place in the table, but no safe generalisation was permissible, because of the omissions and exceptions, until more facts had been collected.

In the search for fresh data I have adopted, with one exception—that of the halogens,—the commercial idea of price as a measure of plenty or rarity. Thus, with current prices, a given sum of money will purchase 1 ounce of gold, 42 ounces of silver, and 2286 ounces of copper—a clear indication that gold (with the highest atomic weight)

* Read before the British Association (Section B), Belfast Meeting, 1902.

is the rarest, while copper (with the lowest atomic weight) is the most plentiful member of this group. The above numbers are here termed the relative distribution figures. The method has its limitations, for the relative distribution figure may vary with the difficulty of isolation of an element, and when not isolated on the proportion of the atomic weight to the weight of the rest of the compound or on the relative money value of the atomic weight of the element to that of the rest of the compound. Thus difficulties of isolation place calcium out of its proper order with barium and strontium; while, in the case of mercury, its high atomic weight and money value, and the comparatively low weight and cheapness of other elements with which it may be combined, as well as its ease of isolation, give it a distribution figure of small variation. Iron, for contra reasons, has a distribution figure of wide variability. In constructing the accompanying table, therefore, it has been necessary in many cases—(1) To choose the commonest forms of combination; (2) to take the value of the combined element, which is at the same rate as the compound itself; and (3) to have the compared elements in the same natural group and combined, as far as possible, with the same simple or compound radical. Disturbing influences outside those of commercial demand are thus in a great measure eliminated, while slight fluctuations in price do not materially alter the order of distribution in any group. The unit of relative distribution is one ounce of gold; in other words, the relative distribution figures are the Troy weight in ounces purchasable with four guineas (£4 4s.), the present price of one ounce of gold. Indication is also given as to what the figure has been calculated from, whether the element R or some compound of it. Blanks are left in places for obvious reasons; but, in the case of gallium, the space is not filled in because this element has become commercially extinct—the material containing it can no longer be obtained. My friend Mr. F. W. Branson, of Leeds, informs me that a grm. of gallium alum is quoted at 350 marks—or, say, a thirtieth of an ounce Troy, for £17. This gives an abnormal relative distribution figure of 0.007, which I have omitted from its group.

Element R.	Atomic weight.	Relative distribution. (From R).
Cu ..	63	2286
Ag ..	108	42
Au ..	197	1
Na ..	23	188
K ..	39	26
Rb ..	85	0.065
Cs ..	133	0.029
(From RCO_3).		
Ca ..	40	7349
Sr ..	87	2450
Ba ..	137	3675
From—		
Mg ..	24	7349 ($\text{RSO}_4 \cdot 7\text{H}_2\text{O}$)
Zn ..	65	3675 ($\text{RSO}_4 \cdot 7\text{H}_2\text{O}$)
Cd ..	112	204 (RSO_4)
Hg ..	200	350 (R)

Element R.	Atomic weight.	Relative distribution. (From R).
Al ..	27	175
Ga ..	70	—
In ..	114	0.16
Gd ..	156	0.03
Tl ..	204	13.8
Ti ..	48	0.58
Zr ..	90	0.52
Ce ..	140	0.20
Th ..	232	0.75
C ..	12	—
Si ..	28	—
Sn ..	119	490
Pb ..	207	1469
N ..	14	—
P ..	31	350
(From R_2O_3).		
As ..	75	1837
Sb ..	120	612
Bi ..	208	122

Element R.	Atomic weight.	Relative distribution. (From R).
O ..	16	—
S ..	32	4899
Se ..	79	12.7
Te ..	127	3
Fe ..	56	1837
Rh ..	102	0.23
Os ..	191	0.35
Ni ..	58.7	188
Ru ..	103	0.27
Ir ..	193	0.50
Co ..	59	72
Pd ..	106	0.43
Pt ..	194	0.88

Element R.	Atomic weight.	Relative distribution. (From $\text{RSO}_4 \cdot 7\text{H}_2\text{O}$).
Fe ..	56	4899
Ni ..	58.7	597
Co ..	59	245

All, except the terminal members in some of the groups, show an inverse relation of atomic weight to relative distribution. In the case of the halogens, it is possible to get an approximation to their *absolute* distribution. Such an estimation is based on the following considerations:—Haloid compounds are admitted by geologists to have been amongst the last to form or condense on the earth's surface. Solvent denudation for untold ages has effectually carried them to the sea, which has been their great receptacle, and it is a reasonable assumption that what is left on the land is a negligible quantity when compared with the vast mass existing in the sea. On this subject wrong ideas are prevalent, based principally on river analyses. It is only necessary for me to point out that the annual fluvial load of haloid compounds is mainly circulatory, and that the portion derived *de novo* from the earth is probably not more than 8000 millionths of what is already in the ocean (Ackroyd, *CHEMICAL NEWS*, lxxiii., 268; *Geol. Mag.*, October, 1901, p. 448; and *Proc. Yorks. Geol. and Polytechnic Society*, xiv., 411). Given, then:—(1) The ratio of chlorine to bromine and of chlorine to iodine in the sea, and (2) the mass of its chlorine contents, one may compute what is their respective per cent of the earth's mass. Adopting the most recent data for all weights and measurements concerned, I get the following figures:—

R.	Atomic weight.	Relative distribution in per cent of the earth's mass.
Chlorine	35.5	0.000486
Bromine	80	0.00000383
Iodine	127	0.0000000583

Or we may express the results as follows:—

a.	b.	c.	$\frac{c}{b}$
R.	Atomic weight.	Per cent of earth's mass.	Atomic distribution.
Cl..	35.5	4.86×10^{-4}	0.136×10^{-4}
Br .	80	5.83×10^{-6}	0.0728×10^{-6}
I ..	127	5.83×10^{-8}	0.0459×10^{-8}

An inverse relation of distribution to atomic mass is strikingly apparent.

The exceptions to this rule occur at the two extremes—among the lightest non-metals and the heaviest metals. As such a law will probably embrace all the elements in the universe, and certainly not less than exist in the solar system, it is within the bounds of probability that the lighter non-metallic elements, absent in requisite proportions in the earth, may exist to a preponderating extent in the outer planets and be a cause of their marked lesser density. This view has already been advocated by Sir J. N. Lockyer, on other grounds entirely, in the course of his Manchester lectures in 1876 ("Science Lectures for the People," Eighth Series, p. 160, John Heywood).

The anomalous position of some of the metals of highest atomic mass is illustrated by the case of barium. This element is more plentiful than its immediate predecessor, strontium—a peculiarity also possessed by mercury, lead, thallium, osmium, platinum, iridium, and thorium. If, however, the distribution figures be divided by the respective atomic weights, mercury, barium, osmium, and iridium cease to be anomalous; platinum is only just over the dividing line, while lead, thallium, and thorium still remain exceptions which might perhaps conform to rule if one could only become possessed of their absolute distribution figures.

In addressing this Section in 1886, Sir William Crookes conjured up the beginnings of things before the earth was thrown off from the central nucleus—a primal stage of ultra-gaseous matter, a vast sea of incandescent mist—when the atoms could not yet have been formed. In time, by some process akin to cooling, this primordial matter (protyle) formed itself into atoms. Does it not now appear that during this genesis of the elements—this transmutation of protyle into complex elemental forms—less and less atoms of highest mass were evolved, and that the

universe became pervaded by the greatest quantity of those atoms which have the lowest masses?

APPENDIX.

Ratio of the Halogens in the Sea.—The extreme values for Cl:Br are 100:0.32 from C. J. S. Makin's "Analysis of the Atlantic" (*CHEMICAL NEWS*, lxxvii., 155), confirming Dittmar's *Challenger* results, and 100:2.11 from Usiglio's figures for the Mediterranean ("Watts's Dictionary," v., 1017). The mean Cl:Br is 100:1.2. Gautier found 2.4 m.grms. of I per litre in sea-water (*Comptes Rendus*, 1899, cxxviii., 1069). Taking the Cl to be roundly 20,000 m.grms. per litre, we obtain the ratio Cl:I = 100:0.012, which is nearly identical with the ratio obtained from Sonstadt's earlier data (*CHEMICAL NEWS*, xxv., 196, 231, 241).

Earth's Mass.—The mean density is taken as 5.472, the average of eight determinations by Maskelyne, Cavendish, Reich, Baily, Airy, James, Poynting, and Plana and Carlini; and the mean diameter as 7912.4 miles, which gives a mass of 5812.4×10^{18} tons.

Mass of Chlorine in the Ocean.— 28286×10^{18} tons.

THE COLOUR OF IODINE-CONTAINING COMPOUNDS.*

By Miss IDA SMEDLEY.

PROFESSOR H. E. ARMSTRONG (*Proc. Chem. Soc.*, 1892) has for some years contended that a generalisation may be made as to the molecular structure of substances which are visibly coloured in the conventional acceptance of the term, since almost all coloured organic substances may be represented by a "quinonoid" structure, if the term "quinonoid" be regarded as including any compound, which contains at least three centres, having some influence on the passage of light through the molecule. From this point of view he regards iodoform as a quinonoid substance in which the three iodine atoms act as centres co-operating to produce colour.

Substances containing iodine may be divided into two classes—coloured and colourless. Does any distinctive similarity of structure exist in the coloured iodine-containing compounds?

Of the molecular condition of solid iodine nothing is known; in solutions the number of atoms in the molecule probably lies between two and four (Loeb, *Trans. Chem. Soc.*, 1888). The violet vapour up to 700° C. consists of di-atomic molecules; but completely dissociated or gaseous mon-atomic iodine is described as colourless. The iodine halogen compounds are also coloured, the mono-chloride much more intensely than the tri-chloride. When iodine acts as a trivalent element, combination with one chlorine atom is insufficient to produce colour: thus di-phenyl iodonium chloride (IPh_2Cl) is colourless, but phenyl iodide chloride (IPhCl_2) is yellow.

Since the iodine atom itself is probably colourless, the colour of the compounds must be attributed to the mutual effect of the halogen atoms. Further, a comparison of the iodine chlorides shows that the tendency to produce colour is greater where the iodine atom is not fully saturated.

Amongst the compounds of iodine with other elements, the readiness with which double iodides are formed, the comparative insolubility of many of the iodides, and the colour-changes they undergo on heating, suggest that their molecular structure is more complex than is usually represented. Nearly all the mono and divalent elements give colourless iodides, and it is probable that the coloured di-iodides are really polymerised. Mercuric iodide, for instance, which exists in two coloured isomeric forms, gives a colourless vapour, the density of which

* Read before the British Association (Section B), Belfast Meeting, 1902.

corresponds with the simple formula HgI_2 . Among organic substances the exceptional case of di-iodoacetic acid and its salts (Perkin and Duppa, *Annalen*, 185), which are all described as yellow, needs further investigation.

In the case of tri-iodides and higher iodides generally, the appearance of colour is determined, not only by the number of iodine atoms present, but also by the condition of the iodine. Thus the nitrogen group of elements form deeply coloured tri-iodides, while the tri-iodides of boron and aluminium are colourless. In hydrocarbon derivatives the three iodine atoms must be associated with the same carbon atom in order to produce colour, as in iodoform; poly-iodo-derivatives in which the iodine atoms are joined to different carbon atoms, as in tri-iodo-benzene, being colourless.

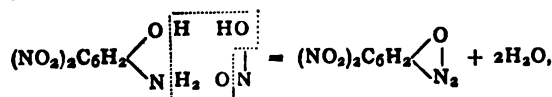
OUR PRESENT KNOWLEDGE OF AROMATIC DIAZO-COMPOUNDS.*

By GILBERT THOMAS MORGAN, D.Sc., F.I.C.

THE aromatic diazo-compounds, which were originally discovered by the classical investigations of Griess (*Annalen*, 1856, cvi., 123; 1860, clxiii., 201; 1866, cxxxvii., 39), have proved to be substances of the utmost value in the development of synthetical chemistry, both from the scientific and the industrial standpoints. The starting-point of these far-reaching researches was a comparative study of asparagine and picramic acid. These substances, which in 1858 were assumed to be compounds of a similar type, behaved quite differently towards nitrous acid, asparagine losing the whole of its nitrogen, whilst picramic acid exchanged three of its hydrogen atoms for one of nitrogen, giving rise to the earliest known diazo-compound—namely, dinitrophenoldiazo-oxide.

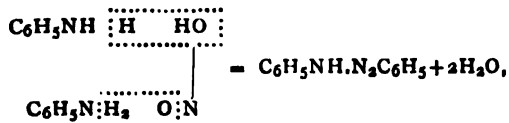
It is interesting to notice at this stage that, although the elimination of aminic nitrogen by the action of nitrous acid is characteristic, not only of asparagine, but also of the majority of aliphatic amino-compounds, yet the preparation of aliphatic diazo-derivatives has been accomplished, and the new field opened up by the discovery of ethyldiazo-acetate (Curtius, *Ber.*, 1883, xvi., 2230) has yielded a rich harvest, culminating in the isolation of hydrazine, hydrazoic acid, and diazo-methane. These important contributions to the chemistry of nitrogen will not, however, be further discussed in this report, which is restricted to a consideration of the aromatic diazo-compounds—a class of substances derived from the hydrocarbons of coal-tar.

The action of nitrous acid on picramic acid is now interpreted in the following manner:—



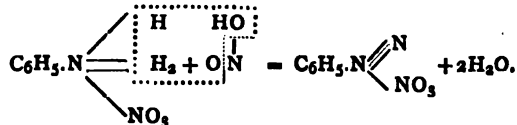
the product being regarded either as the internal salt or the *eso*-anhydride of dinitrophenoldiazo-hydroxide, $(NO_2)_2C_6H_3(OH)N_2OH$ (Hantzsch, *Ber.*, 1896, xxix., 1526).

Griess next extended the investigation to aniline, with the result that diazoaminobenzene was produced,—



* Report presented to Section B, British Association, Belfast Meeting, 1902.

and a subsequent modification of the experimental conditions led to the discovery of benzenediazonium nitrate,—



These three substances, the *eso*-anhydride, the diazo-amine, and the diazonium salt, each containing a diazo-complex, N_2 , attached to one aromatic nucleus, are typical examples of three important classes of aromatic diazo-compounds.

The process of converting the salt of a primary aromatic amine into the corresponding diazonium derivative is termed diazotisation, and this important operation may be suitably considered under a separate heading.

I. THE PREPARATION AND PRACTICAL APPLICATION OF DIAZO-COMPOUNDS.

A. The Diazo reaction.

Benzenediazonium nitrate, the substance formed by the action of nitrous fumes on a cold aqueous solution of aniline nitrate, was found to be a highly explosive salt readily soluble in water or alcohol. On account of these properties, the isolation of the salt and the corresponding chloride and sulphate is a somewhat difficult and dangerous operation, and for many years the properties of these substances were studied in the solutions obtained by the action of the alkali nitrites on the primary aromatic base dissolved in excess of cold dilute acid. It has been found by colorimetric and electrolytic determinations of the velocity of this reaction that, in the absence of disturbing influences, all the aromatic amines are diazotised at approximately the same rate (Hantzsch and Schumann, *Ber.*, 1899, xxxii., 1691; 1900, xxxiii., 527).

It is, however, sometimes necessary to operate with the dry diazonium salts, and these may be prepared by the action of amyl nitrite on solutions of the salts of the amines in absolute alcohol (Knoevenagel, *Ber.*, 1890, xxiii., 2094; Bamberger, *Ber.*, 1896, xxix., 446), or glacial acetic acid (Hantzsch, *Ber.*, 1897, xxx., 92; 1901, xxxiv., 3337).

Excess of acid should be avoided in these preparations, for the diazonium chlorides derived from the halogen-substituted anilines combine with hydrogen chloride, forming acid salts of the types RN_2Cl , HCl , and $3RN_2Cl.HCl$ (*Ber.*, 1897, xxx., 1148 and 1153), these products being less stable than the normal salts themselves.

Bamberger showed that pure benzenediazonium chloride, when dissolved in water, gives a neutral reaction, and differs in this respect from aniline hydrochloride, the solution of which is acid, owing to the hydrolytic dissociation of the salt. This result indicates that the strength of the diazonium base is much greater than that of the corresponding primary aromatic amine.

Griess found that the introduction of a diazonium salt into the alkaline solution of a phenol resulted in the formation of an intensely coloured substance containing the diazo-complex, $N:N$, attached to a second aromatic nucleus,—



These products, which are called azo-compounds, are also produced, either by the reduction and coalescence of two molecules of a nitro-compound, or by the condensation of the nitroso-compounds with the primary amines (C. Mills, *Trans.*, 1895, lxvii., 925).

On account of their condensation to form azo-compounds and their ready reduction to hydrazines, the diazonium salts were for many years assumed to have the constitution $R.N:N.Cl$, a formula which was suggested by Kekulé, although Strecker, Erlenmeyer, and Blomstrand had at different times advocated the claims of the symbol—



According to the former view, which generally prevailed until 1894, the diazonium bases are substances of the oxime type, $RN:NOH$; and this formula, which satisfactorily accounts for the fact, first noticed by Griess, that benzenediazonium hydroxide forms a potassium derivative, is not opposed to the general behaviour of these bases and their salts when employed in the various synthetical operations which engrossed the attention of investigators in this field until the close of the period now under review.

The synthetical application of the diazonium salts will be considered in the next section, since it does not necessarily involve any discussion as to their precise configuration.

B. Diazo-compounds as Agents in Chemical Synthesis.

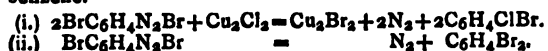
1. Replacement of NH_2 by Cl, Br, I, CN, CNO, or CNS.

—Griess's researches on the production of the halogen derivatives of the aromatic hydrocarbons are now only of historical interest, since the methods commonly employed at the present time are due to Sandmeyer (*Ber.*, 1884, xvii., 2650) and to Gattermann (*Ber.*, 1890, xxiii., 1218; 1892, xxv., 1074). The latter of these investigators also extended the scope of this synthetical process by demonstrating that the cyano (CNO) and thiocyno (CNS) radicals could also be introduced into the aromatic nucleus by the use of the appropriate diazonium salt.

The course of the reaction between diazonium derivatives and cuprous salts (Sandmeyer) or copper powder (Gattermann) has been recently reviewed by Hantzsch (*Ber.*, 1900, xxxiii., 2544), whose conclusions may be summarised as follows:—

The course of the Sandmeyer reaction is not a simple one, and the final result is due to the simultaneous effect of three concurrent actions:—(i.) The formation of a labile diazonium cuprous double salt, which subsequently undergoes decomposition in such a manner that the radicle originally attached to copper migrates to the aromatic nucleus; (ii.) a catalytic action, which becomes the main reaction when copper powder is employed, whereby nitrogen is eliminated from the diazo-salt, so that the acid radicle becomes directly attached to the aromatic nucleus; (iii.) the formation of azo-compounds, which is accompanied by the oxidation of the cuprous salt to the cupric condition.

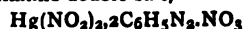
The concurrent effect of the first two reactions was demonstrated by subjecting dry p -bromobenzenediazonium bromide to the action of cuprous chloride dissolved in methyl sulphide. The product consisted chiefly of p -bromochlorobenzene mixed with a little p -dibromobenzene.



Cuprous bromide and p -bromobenzenediazonium chloride yielded p -dibromobenzene containing a little p -bromochlorobenzene. In both cases the first reaction predominates, and it may, under certain conditions, prevail to the almost complete exclusion of the second. Cuprous iodide, for example, gave rise to iodo-derivatives only, with various diazonium chlorides and bromides, and, on the other hand, the interaction of cuprous chloride and benzenediazonium iodide furnished chlorobenzene unaccompanied by iodobenzene.

2. Replacement of NH_2 by NO_2 .—The introduction of nitroxy through the agency of the diazonium salt was first indicated by Sandmeyer, and further exemplified by Hantzsch (*loc. cit.*) in the following experiments:—

(i.). The crystalline double salt,—



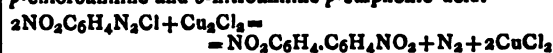
(melting-point 76°), obtained by mixing solutions of benzenediazonium nitrate and potassium mercuric nitrite, decomposes on boiling with water, yielding phenol and nitrophenol, but when treated with copper powder it furnishes a quantitative yield of nitrobenzene. (ii.). The diazonium sulphates, when mixed with a freshly prepared

suspension of cupro-cupric sulphate and treated with excess of an alkali nitrite, give rise to the corresponding nitro-derivatives; 2 : 4 : 6-tribromobenzenediazonium sulphate, for example, gives a 65 per cent yield of 1-nitro-2 : 4 : 6-tribromobenzene.

β -Nitronaphthalene, a substance prepared with considerable difficulty by other processes, is produced from β -naphthylamine to the extent of 25 per cent by this method, whereas Sandmeyer, who employed cuprous oxide and the diazonium nitrite, obtained only 7 per cent (*Ber.*, 1887, xx., 1497).

3. Formation of Conjugated Systems $R \cdot R$ or $R \cdot N : N \cdot R$.

—The third reaction signalled by Hantzsch may be rendered more apparent by reversing the usual order of mixing and adding the cuprous chloride dissolved in hydrochloric acid to the cold solution of the diazonium salt. Under these conditions, aniline, o -chloroaniline, and the o - and p -toluidines give rise to appreciable quantities of azo-compounds. The nitrated amines, however, behave very differently, yielding diphenyl-derivatives; this result being obtained with the three nitranilines, o -nitro- p -chloroaniline and o -nitroaniline- p -sulphonic acid.



(Ullmann and Forgan, *Ber.*, 1901, xxxiv., 3802; Niementowski, *Ber.*, 1901, xxxiv., 3325).

This type of condensation is also brought about by the use of cuprous oxide in ammoniacal or hydroxylamine solution (*Annalen*, 1902, cccxx., 122).

4. Introduction of the Sulphonic Radicle SO_3H .—The replacement of NH_2 by SH was first accomplished by Leuckart (*Journ. Prakt. Chem.*, [2], xli., 218), who treated the diazonium salt with an alkali xanthate and hydrolysed the resulting aromatic xanthate, thus obtaining either the thiophenol or the disulphide produced by oxidation. Either of these products yields the corresponding sulphonic acid on treatment with alkaline permanganate solution (F. Bayer and Co., *D.R.P.*, 70286; Wynne and Bruce, *Trans.*, 1898, lxxiii., 738).

The production of sulphinic acids by the direct action of sulphurous acid on diazonium salts appears to have been first observed by Müller and Wiesinger (*Ber.*, 1879, xli., 1348). A simple process, due to Gattermann (*Ber.*, 1899, xxxii., 1136), was accidentally discovered during an investigation of o -methoxybenzenediazonium chloride, when it was found that this salt on treatment with sulphurous acid yielded a diazonium sulphite which, on mixing with copper powder, evolved nitrogen, giving rise to the corresponding sulphinic acid; this product being subsequently oxidised by means of permanganate solution.

In general, the reaction takes place most readily with the diazonium sulphate, a cold solution of this salt in a large excess of dilute sulphuric acid being saturated with sulphur dioxide, and finally treated with copper powder.



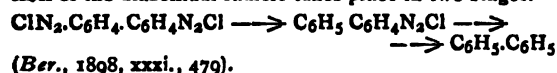
In the case of the diazotised naphthylamines it is better to add their solutions to the mixture of reduced copper and sulphurous acid.

These processes for the production of sulphonic acids have not, however, been successfully applied to the diazonium salts derived from the nitrated aromatic amines.

5. Replacement of the Diazo-radicle by Hydrogen.—The method originally employed for the elimination of the diazo radicle consisted in boiling the diazonium chloride with absolute alcohol; this operation does not, however, invariably give the required result, and Hantzsch (*Ber.*, 1901, xxxiv., 3337) has accumulated evidence in support of the view that the normal decomposition of a diazonium salt by an alcohol leads to the production of an alkyl phenoxide. Benzenediazonium chloride or sulphate when boiled with methyl alcohol yields anisole unaccompanied by benzene. Increase in the molecular weight of the alcohol or the accumulation of negative substituents in

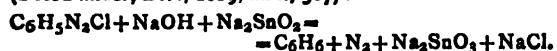
the aromatic nucleus diminishes the yield of ether and augments that of the hydrocarbon. The benzene-diazonium salts, when treated with ethyl alcohol, yield phenetole and a trace of benzene; their chloro- and bromo-derivatives, when boiled with this reagent, give halogenated benzenes but no substituted ethers, whereas methyl alcohol converts them into mixtures consisting chiefly of the substituted phenoxide and a small quantity of the halogenated hydrocarbon. Numerous examples of this reaction will be found in the work of Remsen and his pupils (*Amer. Chem. Journ.*, xv., 105; xix., 531, 547, 561).

In the case of diphenyltetrazonium chloride the elimination of the diazonium radicle takes place in two stages.



The reversion to the parent hydrocarbon is more readily effected by the process introduced by Baeyer and Pützinger (*Ber.*, 1885, xviii., 90, 786; compare Wynne and Bruce, *loc. cit.*), which consists in reducing the diazonium salt to the hydrazine with stannous chloride, and subsequently removing the hydrazino-radicle NH.NH_2 by boiling with cupric sulphate solution.

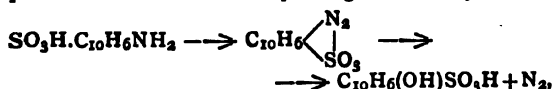
Sodium stannite has been recommended as an agent for reducing the diazonium salt to the hydrocarbon (Friedländer, *Ber.*, 1889, xxii., 387):—



Mai has recently found that β -toluenediazonium chloride, when added to a strong solution of hypophosphorous acid, gives rise to toluene, the yield being 67 per cent. Benzenediazonium chloride yields a mixture of benzene (two parts) and diphenyl (one part), whilst diazotised benzidine and α -naphthylamine furnish diphenyl and naphthalene respectively (*Ber.*, 1902, xxxv., 162).

6. *Substitution of NH_2 by OH .*—The replacement of NH_2 by OH , although an extremely important synthetical operation, can hardly be included amongst the modern developments of the application of diazonium salts, inasmuch as the process still employed, which consists in boiling the aqueous solution of the diazonium salt, is a legacy derived from Griess's pioneering researches.

The manufacture of the 1:4- and 1:8- α -naphtholsulphonic acids from the corresponding amino-compounds—

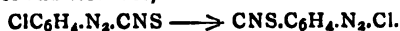


and the production of 1:8-dihydroxynaphthalene 3:6-disulphonic acid (chromotrope acid) may be cited as examples of the application of this process.

7. *Miscellaneous Substitutions.*—There are several other modes of reaction which, although of less importance from the synthetical point of view, are nevertheless of interest as indicating the extremely reactive character of the diazonium salts.

(i.). The amino groups in certain azo-derivatives of β -naphthylamine are replaced by the acetoxy-radicle when these substances are diazotised in warm glacial acetic acid (Meldola and East, *Trans.*, 1888, liii., 460) and Orndorff (*Amer. Chem. Journ.*, 1888, x., 368) showed that this reaction may be generally employed in the production of aromatic acetates.

(ii.). A remarkable example of intramolecular change was noticed by Hantzsch in studying the chloro- and bromo-diazonium thiocyanates (*Ber.*, 1896, xxix., 947; 1898, xxxi., 1253). These salts, when dissolved in alcohol containing a trace of hydrochloric acid, become converted into the isomeric thiocyanobenzenediazonium chlorides and bromides,—



This change takes place only when the halogen atom is

situated in an ortho- or para-position with respect to the diazonium group; transformation does not occur in the case of *m*-chlorobenzenediazonium thiocyanate.

The extent to which this rearrangement is possible is best indicated by the limiting case, 2:4:6-tribromobenzenediazonium sulphate in the presence of excess of potassium thiocyanate actually giving rise to 2:4:6-trithiocyanobenzenediazonium thiocyanate $(\text{CNS})_3\text{C}_6\text{H}_2\text{N}_2\text{CNS}$.

(iii.). Another extremely interesting case of molecular rearrangement is the conversion of the bromodiazonium chlorides into the isomeric chlorodiazonium bromides (Hantzsch, *Ber.*, 1897, xxx., 2334; 1900, xxxiii., 505; and *Journ. Prakt. Chem.*, xxvii., 98).

This transformation, which has been studied quantitatively, is a mono-molecular change, subject to the following laws:—

1. The bromine atoms undergo replacement only when present in the para- or ortho-position with respect to the diazo-radicle, those in the ortho-position being most readily removed. Metabromo-derivatives are not affected.

2. The ease of transformation increases with the number of bromine atoms present.

3. The transformation constant, calculated from the equation $x = 1/t \log a/(a-x)$, increases with the temperature, and is also influenced by the solvent, having its minimum value in water, and becoming greater as the series of alcohols is ascended.

The diazonium salts containing two bromine atoms are stable when dry, but rapidly undergo conversion in ethyl alcohol; 2:4:6-tribromobenzenediazonium chloride becomes transformed even in the dry state.

(iv.). Dibenzoylhydrazines $\text{RN}(\text{COC}_6\text{H}_5)_2\text{N}(\text{COC}_6\text{H}_5)_2\text{R}$ are obtained by treating diazonium salts with an aqueous suspension of benzoyl chloride and copper powder (*Ber.*, 1902, xxxv., 1964).

(To be continued).

THE ALKYLATION OF SUGARS.*

By THOS. PURDIE, F.R.S., and JAMES C. IRVINE, B.Sc., Ph.D.

THE method of alkylating hydroxyl groups by means of a mixture of silver oxide and alkyl iodides does not appear directly applicable to aldoses or ketoses, and results in oxidation and subsequent changes of some complexity. By means of this reaction, however, the authors have succeeded in alkylating methyl glucoside with the formation of tri-methyl and tetra-methyl methyl glucoside ethers, compounds from which alkylated aldoses can presumably be obtained on hydrolysis.

Tri-methyl Methyl Glucoside Ether.—The methyl glucoside used in the preparation was obtained by heating glucose in CH_3OH solution with HCl for fifty hours at 100° . The glucoside (melting-point $165-167^\circ$) was dissolved in boiling methyl alcohol, and a large excess of the alkylating mixture, in the proportion of two molecules iodide to one of oxide, gradually added. After all action ceased, the mixture was boiled under a condenser for over an hour, and filtered.

On removal of the alcohol, the filtrate was extracted with ether, and after drying and evaporation, the neutral syrupy extract was distilled in vacuum from a graphite bath. An extremely thick colourless liquid passed over between $155-157^\circ$ under 12 m.m. pressure, and after refractionation a sample gave on analysis figures in close agreement with the calculated numbers for tri-methyl methyl glucoside ether:—



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* Read before the British Association (Section B), Belfast Meeting, 1902.

A molecular weight determination showed that no rupture of the molecule had occurred, and the methoxyl content of the compound was confirmed by Zeisel determinations.

The higher fractions obtained in the vacuum distillation were extremely thick, and the results of analyses showed these to consist of incompletely methylated glucoside.

Tri-methylic methyl glucoside is optically active, a 5 per cent solution in alcohol showing a specific rotation of $+126.75^\circ$ at 20° , whilst for the pure substance $[\alpha]_D^{30} = +129.27^\circ$. The compound is soluble in water, alcohol, ether, and methyl iodide, and the aqueous solution is without action on Fehling until hydrolysed with HCl, when the aldose produced in the reaction effects ready reduction.

Tetra-methylic Methyl Glucoside.—This compound was prepared by adding silver oxide to a solution of the tri-methylic ether in a large excess of methyl iodide, and boiling the mixture for several hours. After filtering and evaporating, the product was distilled in vacuum, and tetra-methylic methyl glucoside obtained as a colourless fairly mobile liquid, boiling at $144\text{--}145^\circ$ under 17 m.m. pressure. In different preparations the boiling-point was the same, and satisfactory results were obtained in the combustions and Zeisel determinations. The specific rotation at 20° of the pure substance proved to be $+127.88^\circ$, whilst a 5 per cent solution in alcohol gave $[\alpha]_D^{20} = +127.78^\circ$.

Hydrolysis of the Compounds.

Both tri-methylic and tetra-methylic methyl glucoside when hydrolysed give compounds capable of reducing Fehling's solution, and therefore probably containing an "aldose" structure. On hydrolysis of the tri-methylic ether by means of 20 per cent HCl, a colourless gum was isolated from the reaction product, which, after drying at 110° , gave figures on combustion approximating to those required for tri-methyl glucose. The substance reduced both Fehling's solution and an ammoniacal solution of silver nitrate, and a qualitative Zeisel experiment showed that the methoxyl groups had survived the hydrolysis. So far this body has not been obtained in a state of purity, but we are at present engaged in the preparation of the compound on a larger scale, with a view to examining its properties and investigating the special reactions of which it is capable. We likewise hope to publish shortly an account of the alkylated glucose obtainable from the tetra-methylic methyl glucoside, and to continue our experiments on other alkylated sugars which are already partly prepared and examined.

Alkylation of Acetone-rhamnoside.

According to Fischer's formula for acetone-rhamnoside, the compound contains two hydroxyl groups, and we find that the silver-oxide reaction can be successfully carried out on the substance with the formation of di-methylic acetone-rhamnoside.

The rhamnoside, when dissolved in acetone, reacted energetically with silver-oxide and methyl iodide, and after filtration and evaporation the product was distilled in vacuum. Complete alkylation was, however, only obtained by dissolving the distilled product in methyl iodide and repeating the treatment with silver-oxide. On distillation, a colourless pleasant-smelling oil was obtained (boiling-point $= 115\text{--}118^\circ$ under 11 m.m. pressure), which the combustion and Zeisel results showed to be di-methylic acetone-rhamnoside.

The compound is soluble in the ordinary organic solvents, and has scarcely any action on Fehling until hydrolysed with HCl.

A five per cent solution in acetone had a specific rotation of -24.64° at 20° , this result being in sharp contrast to the value of $[\alpha]_D$ for the parent substance, which equals $+17.4^\circ$.

Presumably the compound on hydrolysis splits off a molecule of acetone and gives a di-methylic rhamnose just as the rhamnoside itself yields acetone and rhamnose.

We are at present engaged in the investigation of this reaction, and it is our intention to examine the hydrolysis product in detail, with a view to establishing its reactions and constitution.

ON SUB-OXIDE OF LEAD.

By R. W. EMERSON MACIVOR, F.I.C., &c.,
Formerly Lecturer on Chemistry, Anderson's College, Glasgow.

REQUIRING some sub-oxide of lead in connection with an investigation in which I have been engaged, I adopted the method of making it used by Dulong, Boussingault, and Pelouze, viz., carefully heating lead oxalate in a retort from which air is excluded, but found the product invariably contain a greater proportion of lead than theory required for the sub-oxide. Thus, in four different specimens the lead ranged from 96.89 to 98.36 per cent, theory demanding 96.28 (Pb=207). These irregularities in composition were undoubtedly due to the reduction of sub-oxide by carbon monoxide in the atmosphere of the retort.

Pelouze states that the oxalate should not be heated to a higher temperature than 300° , the heat being continued as long as any gas is given off. He found the gas thus evolved to consist of a mixture of CO and CO_2 in the proportions of 1 : 3 [$2\text{PbC}_2\text{O}_4 = \text{Pb}_2\text{O} + \text{CO} + 3\text{CO}_2$], excepting towards the end of the operation, when, particularly if "the heat be somewhat increased in order to finish the decomposition, the gas is richer in CO_2 ." This increase in the proportion of CO_2 can, of course, only arise from the reduction of Pb_2O .

I find that if a heated current of pure carbon dioxide be maintained through the retort during the decomposition of the lead oxalate so as to rapidly remove the CO immediately it is formed, and the temperature not allowed to exceed 300° , but kept rather a little under that point if anything, the final product is true sub-oxide free from PbO and metallic lead. It is a dull black powder tinged with grey, which bears lengthened exposure to dry air without undergoing change. Heated strongly— 350° or so—it alters in colour to greenish grey, being split up into Pb and PbO. It is not decomposed by water, but is acted upon by dilute alkalis and acids, and resolved into metal and PbO.

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THE LITMUS-PAPER TEST FOR MILK.

By H. DROOP RICHMOND, F.I.C.

In early text-books of chemistry an acid was wrongly, as we now know, defined as a substance reddening blue litmus-paper, and an alkali as a substance which turned red litmus-paper blue. An indicator is really a coloured salt of an acid having an ion of a different colour, and the behaviour of acids towards indicators depends on the relative dissociation constants of the acid and the indicator.

Indicators may be divided into classes, those which approach the limit of complete dissociation in a solution dilute enough to show the colour, those which approach the limit of no dissociation, and those intermediate, or, in other words, those whose acids are strong, weak, and medium.

Litmus is an indicator of the medium class, i.e., it is unaffected by weak acids, and incompletely affected by acids of medium strength.

Litmus-paper may be either red containing only the acid, or blue containing the acid with such an amount of alkali that no red ions are formed, or at some intermediate stage. If we test with these papers a partially neutralised mixture of acids of various strength, we may obtain quite contradictory results.

Phosphoric acid is a good example of three different acidities in one molecule; the first acidity is strong, the third is very weak, and the second is intermediate between the two, and about equal in strength to the acid of litmus. It has been shown that milk contains phosphates with the third acidity completely neutralised, and the second only partly so, and therefore milk is an excellent substance to show the peculiar behaviour of litmus.

If we take a blue litmus-paper and dip it into milk, the blue litmus having the acid completely neutralised is more alkaline than the milk, and the two tend to come into a condition of equilibrium by a portion of the alkali of the litmus passing to the milk; the consequence is that the litmus becomes less alkaline, and turns slightly red. If we take a red litmus-paper, which is more acid than the milk, alkali will tend to pass from the milk to the litmus, and turn it slightly blue. This is the so-called amphoteric reaction. A litmus-paper of some intermediate stage would be unaffected.

It is therefore quite fallacious to try and test the acidity of milk with litmus-paper; the old test to see if milk is sour by putting in a piece of blue litmus-paper has long been discarded by scientists, but unfortunately is still recommended by those who have the little knowledge which so often proves a pitfall; with a suitable litmus-paper all fresh milk can be condemned by this test, while, by judiciously changing the paper, milk which is on the turn can be *triumphantly proved* fresh.

THE PREPARATION OF GASEOUS PHOSPHIDE OF HYDROGEN.

By F. BODROUX.

AMONG the metallic phosphides, there are a certain number which, when treated with water or acids, give off phosphoretted hydrogen in a more or less pure state. Such is the case with the phosphide of calcium generally used in laboratories, and the same is also true with the phosphides of aluminium and magnesium.

M. Matignon (*Bull. Soc. Chim.*, vol. xxiii., p. 498) made use of the action of dilute acids on phosphide of aluminium for the preparation of gaseous phosphoretted hydrogen. As this latter compound is difficult to obtain we propose to replace it with a less pure compound, but one which has the advantage of being easily prepared in a few moments.

As intimate a mixture as possible is made, consisting of two parts of powdered aluminium and one part of red phosphorus; this can be set light to either directly or by the help of a sheet of paper on which it is placed. The combustion proceeds rapidly, with a brilliant yellow flame; fumes of phosphoric anhydride are given off, and a greyish mass remains behind; this consists of unchanged metal, with oxide and phosphide of aluminium.

When treated with cold water, this substance is decomposed slowly, and gives off pure phosphoretted hydrogen. The reaction is more energetic if we use warm water, but it must not be heated to above 50°, or the product obtained would contain traces of hydrogen. The proportion of this element will be greater as the temperature of the water approaches nearer to the boiling-point.

If an acid solution be used for this operation, the gas, which is given off very abundantly, will consist of a mixture of hydrogen and phosphide of hydrogen, from which the latter body can be separated easily by means of a hydrochloric solution of cuprous chloride.

In the preceding experiment the aluminium can be replaced advantageously by magnesium. A mixture is made of equal weights of magnesium powder and red phosphorus. The operation must be carried out with every care, using only small quantities at a time, or else, on account of the friction, the material might suddenly take fire. The mixture is set alight, and after the com-

bustion is over a yellowish substance remains, which is energetically decomposed by cold water, giving off pure phosphoretted hydrogen.

It is necessary, if we wish to collect the phosphide of magnesium with little trouble, to spread out the mixture of red phosphorus and the metal, in a thin layer over a large surface. The combustion will then take a little longer to be effected, and the loss of a small portion of the compound by projection is prevented.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 12.

THE DETECTION OF VERY SMALL QUANTITIES OF ARSENIC.

By GABRIEL BERTRAND.

In a previous paper on "The Existence of Arsenic in the Organism" (*Bull. Soc. Chim.*, Series 3, vol. xxvii., p. 848) I stated that I had so far perfected the method of detecting arsenic that I could detect a thousandth or even half a thousandth of a milligramme of this metalloid. In this paper I propose to give the details of the method and the precautions it is necessary to take to obtain such a result.

In the first place, the arsenic must be concentrated in a very small volume of liquid. I generally work with such quantities that at the end of the operation there is not more than from 30 to 60 c.c. of liquid in the hydrogen apparatus. It is quite by exception that the volume is sometimes double.

The apparatus must be then entirely freed from all trace of oxygen; without this precaution, a portion, or even the whole of the arsenic set at liberty, becomes oxidised, and passes into the state of arsenious acid, which is much more difficult or even impossible to detect. This is easily effected by forcing through the apparatus, all fitted up and charged with zinc, a good current of pure carbonic acid, such as may be obtained from bottles of liquid carbonic acid. The gas I used contained 0.08 per cent of oxygen. If we prefer to wash out the apparatus with the hydrogen produced therein, we are obliged to leave a notable quantity of acid liquor which will thus dilute the arsenical solution unless special means are taken to prevent this. Much time is saved, however, by proceeding as I have said. With a good current of carbonic anhydride, a few minutes is quite sufficient to get the apparatus perfectly free from oxygen, which result by the old method could not be obtained in less than an hour at the very least.

At the commencement of passing the gas the analysis tube is disconnected so that the gas may pass more easily.

When the apparatus is quite free from air a solution of one or two drops of dilute chloride of platinum in about 10 c.c. of one-fifth sulphuric acid is poured on the zinc, and we wait for about ten minutes before introducing the arsenical liquor.

Small glass tubes very finely drawn out are used as analysis tubes. If, for example, we are searching for quantities of arsenic, decidedly less than $\frac{1}{100}$ th of a m.grm., it is advisable to use tubes having an internal diameter of not more than 1 m.m.

After having washed them with warm sulphuric acid or aqua regia, then with water, alcohol, and ether, they are completely dried, and cut to such a length that the end will be from 10 to 15 c.m. from the place where the ring will be formed; finally, this extremity is drawn out very fine for several centimetres to prevent the diffusion of air into the interior during the operation. If this precaution be not taken, very small deposits of arsenic would become invisible through oxidation as they were produced.*

* This oxidation even takes place with considerable rapidity in the cold, in the tubes in which the rings are kept, if we have not taken the precaution to seal them up full of dry hydrogen. Instead of an easily seen black deposit of the metalloid we only obtain a whitish trace of arsenious acid, sometimes imperceptible even on a black background.

It is not necessary to heat the analysis tube to a higher temperature than nascent redness, nor even, so it seems, along much of its length. However, in nearly all my experiments I used a row of small flames, about 20 cm. in length, in preference to the flat Bunsen flame previously used.

It is advisable to dry the current of gas as it leaves the flask, by passing it through a column of cotton-wool previously heated to 120–120°. The cellulose thus dehydrated eagerly retains the watery vapour without acting on the composition of the gas.

Finally, to prevent the deposit of arsenic from spreading over too great a surface of the analysis tube, which can easily happen with capillary tubes having thick sides, it is as well to encourage the rapid condensation of the arsenical fumes, by surrounding the tube with a small refrigerator at some convenient place.

This latter may consist of simply a band of filter-paper, 4 or 5 mm. in width; this strip of paper is wrapped round the tube two or three times and water falls on it, drop by drop, from a little reservoir above.

The excess of water runs off by the free end. The deposit of arsenic, collected thus in a small space, runs no risk of being overlooked. With quantities of the metalloid of over $\frac{1}{100}$ th of a m.grm., and with tubes having thin sides, the use of the condenser is not necessary.

By taking all these precautions, as well as those already described by A. Gautier (*Ann. Chim. Phys.*, Series 5, vol. viii., p. 384), we are able to obtain a black ring, distinctly visible, of several millimetres in length, with as little as $\frac{1}{100}$ th of a m.grm. of arsenic.

It will be easily understood that with a method of research of such delicacy, it is very difficult to obtain reagents free from arsenic. This is especially true of nitric acid. The purest I have been able to procure commercially, and which was prepared specially for me, still contained nearly $\frac{1}{100}$ th of a m.grm. of the metalloid per kilogram. It was impossible to get completely rid of this arsenic by a series of simple distillations in a glass retort. It was only by three distillations, each time in the presence of one-tenth its weight of pure sulphuric acid, that this proportion could be reduced to less than $\frac{1}{600,000,000}$ th; that is to say, to no longer detect $\frac{1}{100}$ th of a m.grm. in 600 grms. of the acid.*

During the research described above, I was also occupied with the destruction of organic matter. The method which gave me the best results was that of A. Gautier (*Comptes Rendus*, vol. cxxix., p. 936–938). The modifications which it has been recently proposed to make in this method, and which required a large quantity of acid, are unfavourable for the study of the arsenic normal to the organism. As I have made quite certain by direct experiments, and as can be seen from the attempts to purify reagents, there are always traces of arsenic carried over by the acid fumes; so that the modifications proposed, which give complete solutions of the organic matter, and which appear to be very good for the detection of appreciable quantities of arsenic, are of no use for infinitesimal proportions of the metalloid.

For example, on attacking 50 grms. of calves' liver, to which was added $\frac{1}{100}$ th of a m.grm. of arsenic, with 10 grms. of sulphuric acid and 45 grms. of nitric acid in a first experiment, and with 50 grms. of sulphuric acid and 200 grms. of nitric acid in a second experiment, a ring was obtained representing approximately the amount of arsenic added in the first place, while there was nothing, or at best only the most doubtful trace, in the second case.

Instead of using nitric acid alone for the attack, I found it preferable to employ a mixture of 10 parts of nitric and 1 part of sulphuric acid. This mixture penetrates the

carbonaceous mass better, and, not being so volatile as nitric acid alone, it makes it possible, towards the end of the attack, to clean the warm sides of the crucible more easily. With the exception of this slight variation we proceed exactly according to A. Gautier's instructions; there is a little more sulphuric acid in the liquid extract, but that does not offer any inconvenience.

To detect traces of arsenic as small as those mentioned above, only thoroughly well digested products, quite free from organic matter, should be introduced into the Marsh's apparatus; this is a matter of very great importance.

When the precipitate, obtained by the action of the sulphydric gas, gives a brown solution with ammonia, charged with notable quantities of organic matter, the residue left must be attacked by the evaporation of the ammoniacal solution exactly in the same manner as the original material.

If the aqueous solution, separated by filtration from the humic products, remains colourless, or nearly so, it is evaporated to dryness, and the residue dissolved in sulphuric acid at one-fifth is poured into the hydrogen apparatus. If, on the other hand, the aqueous solution is strongly coloured yellow, as happens occasionally when great care has not been taken in the destruction of the organic matter, it is necessary to precipitate the arsenic again by means of sulphuretted hydrogen.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 16.

THE MICROMETRIC ASSAY OF GOLD ORES.

By M. GUERREAU.

It often happens in the assay of poor gold ores, and of slimes, tailings, and other residues from the treatment of auriferous minerals, that the gold bead resulting from the assay, when placed in the balance, is so light that it cannot be weighed with any degree of accuracy. Harkort and Plattner (*Probirkunst*) measured the small beads, obtained by the cupellation of these ores, by means of two lines ruled on ivory similar to those in a Wedgwood pyrometer, and graduated by the help of spheres of gold of known weight.

The results obtained were not very exact, as the beads were flattened on one side and contaminated with silver. Goldschmidt proposed using a micrometer to measure the spheres, and Gozdorf (*CHEMICAL NEWS*, 1886) devised the method of forming the gold into a perfect sphere by fusing it in a bead of boric acid.

I have made a number of assays to check the correctness of this method, which, up to the present, has not been generally made use of in practical assaying.

The gold and silver bead is obtained in the ordinary manner, but the parting with nitric acid must be done with great care, so that the attack may proceed very slowly, and that if the gold is divided it may divide as little as possible. The flattened bead is placed in a small porcelain crucible, and treated with cold nitric acid of 1.18 density (22° B.) for about five minutes; next it is heated gently for ten minutes; then the temperature is raised, and it is treated for ten minutes with nitric acid of 1.29 (32° B.) density. If, at the commencement of the attack, too strong an acid or too high a temperature is used, the gold will be reduced to such a fine state of division that it would be difficult to avoid losses. It is then washed with water, decanted, and dried in the oven.

It is necessary to use a very gentle heat to drive off the last traces of moisture; in fact, if the water is driven off too rapidly, and above all if it is boiled, the gold will be disintegrated, and losses will occur.

As a general rule, inquartation is not necessary, as the silver contained in the assay litharge is amply sufficient.

A borax bead is melted in the blowpipe flame on a platinum wire, and, while still warm, it is pressed on the gold, which adheres to it firmly. On re-melting the bead

* The examination of the acid was effected by evaporating down 300 grms. with 20 grms. of pure sulphuric acid, added gradually, in a porcelain crucible. The evaporation is continued until only about 15 grms. of sulphuric acid are left; this is diluted with four parts of water, and after cooling transferred to the Marsh's apparatus.

the gold collects in the form of a sphere. It sometimes happens that two spheres of gold are obtained instead of one; it is impossible to make these two unite in the interior of the bead; in such a case the boric acid must be dissolved and the operation re-commenced. Although the great viscosity of the melted medium prevents the gold from becoming alloyed with the platinum, it is always advisable to make the borax bead as large as possible.

The boric acid is dissolved, and the sphere of gold measured under the microscope on a watch-glass. An enlargement of 100 to 150 times is most convenient for these assays, but the true value of the divisions of the ocular should be verified by means of a millimetre objective divided into 100 parts.

The sphere should be measured in six or eight different positions, and the mean of the values obtained taken. By operating in this manner, differences in diameter up to 0.03 m.m. have no appreciable influence on the results.

As a general rule, the spheres are very regular; the greatest variations I have observed were on a large sphere of 0.799 m.m. diameter, weighing 5.3 m.grms. The difference between the two extreme measurements were 0.036 m.m., or 4.5 per cent. On small spheres the difference is nil; if, however, too great a variation is found it will be necessary to re-melt the sphere. As a general rule, it may be said that the smaller the sphere the better are the results. It is not advisable to estimate quantities of gold of more than 6 or 7 m.grms. by this method, but on the other hand, there is, so to speak, no limit to the smallness of spheres of which the valuation is possible. The smallest sphere I have obtained weighed 0.04 m.grm. Gozdorf measured the very small spheres in the borax bead, but this method did not give me good results; as a matter of fact, fused boric acid is highly refrangible, and, further, a kind of luminous halo is formed round the sphere of gold, which interferes with the accurate observation of the edges.

The weight of a sphere of a substance of which the density is known is deduced from the formula—

$$W = \frac{\pi \times d^3 \times D}{6},$$

in which d represents the diameter of the sphere, and D the density of the substance; from this we get, $W = 10^{-12} \times d^3 \times D$, a constant with which we determine the weight of a sphere of gold having a diameter of one division of the micrometer. It only remains to multiply the figure thus obtained by the cube of the number of divisions of the sphere to obtain its weight. The sphere may be then weighed on the balance as a check. Gozdorf took 19.5 as the density of gold, but this figure gives results which are about 12 per cent too high. I used the density 19.33, determined by Gustave Rose, and which is generally accepted as the density of fused gold.

The following experiments set out in the accompanying table show the degree of accuracy which can be attained by this method. It will be seen that the differences per cent between the weights found on the balance and those by the micrometer seem considerable, but they are illusory

No.	Diameter in hundredths of a m.m.	Weight on the balance in m.grms.	Micrometric, weight in m.grms.	Difference Per cent.
1.	27.2	0.2	0.204	+2.00
2.	34.5	0.45	0.415	-7.77
3.	56.5	1.9	1.830	-3.68
4.	56.6	1.95	1.836	-5.84
5.	56.9	1.95	1.861	-4.56
6.	60.0	2.2	2.189	-0.50
7.	61.5	2.25	2.351	+4.53
8.	61.9	2.4	2.401	+0.04
9.	62.0	2.5	2.411	-3.56
10.	79.7	5.4	5.117	-5.22
11.	79.9	5.3	5.167	-2.51
12.	82.7	5.75	5.733	-0.33
13.	85.9	6.7	6.407	-4.37

to a great extent, and are due principally to the impossibility of determining the twentieth and even the tenth of a milligramme with certainty on the balance. To prove this, I weighed together the spheres 3, 6, 7, and 8. The weight on the balance was 14 m.grms., the micrometric weight was 13.939 m.grms., or a difference of -0.43 per cent. To these spheres I added Nos. 2, 5, 12, and 13; the weight on the balance was 28.4 m.grms.; the micrometric weight was 28.355 m.grms., or a difference of -0.15 per cent.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 15.

PROCEEDINGS OF SOCIETIES.

MANCHESTER LITERARY AND PHILOSOPHICAL SOCIETY.

Ordinary Meeting, October 7, 1902.

Mr. CHARLES BAILEY, President, in the Chair.

A PAPER ON "The Reaction of Iodine with Mercuric Oxide" was read by Mr. R. L. TAYLOR.

In a former paper the author had shown that, when aqueous iodine is shaken up with precipitated mercuric oxide and rapidly filtered, the filtrate contained 80 to 90 per cent of the possible amount of hypiodous acid. Messrs. Orton and Blackman have stated, in a paper read before the Chemical Society, that the solutions obtained from iodine and mercuric oxide contain only a small quantity of hypiodite, the iodine existing mainly as iodic acid. Mr. Taylor concludes, from the description of these experiments, that Orton and Blackman were not aware of the extremely unstable nature of hypiodous acid. They used ordinary powdered iodine, which is not sufficiently finely divided, and they took a great deal too long over their experiments. Using precipitated iodine, and performing the experiments as rapidly as possible, Mr. Taylor finds that, with ten to twenty-five times as much iodine in proportion to the water as he formerly used, the solution contains from 44 to 52 per cent of the possible amount of hypiodous acid and very little iodic acid—results very different from those of Orton and Blackman.

NOTICES OF BOOKS.

Methods of Gas Analysis. By Dr. WALTHER HEMPEL, Professor of Chemistry in the Dresden "Technische Hochschule." Translated from the Third German Edition, and Considerably Enlarged, by L. M. DENNIS, of Cornell University. London: Macmillan and Co., Ltd. New York: The Macmillan Company. 1902. Pp. 490.

MANY important investigations in the field of gas analysis have been published in the interval that has elapsed since the appearance of the second edition of this work, and it is with the idea of bringing together these and his own experimental researches that the author has been led to prepare this third edition. The work has been thoroughly revised by both the author and the translator, and has been changed to such an extent that this translation may be regarded as a fourth edition of the book.

The additions comprise several new methods of collecting and keeping gas samples; determinations with the Honigsmann, Bunte, and Orsat apparatus; the new Hempel methods for exact gas analysis, new methods for the determination of combustible gases, the separation of argon from the atmosphere, improved methods for the determination of carbon monoxide in gas mixtures, the analysis of acetylene gas, the examination of gases pro-

duced by living bacteria, the simultaneous determination of fluorine and carbon dioxide, the determination of the heating power of gases, sulphur in organic substances, and the volumetric determination of carbon in iron, &c.

The book is divided into three principal parts and eighteen chapters; the third part on "Practical Applications of Gas Analysis" being of the greatest interest to analytical chemists, as it deals with the definite technical work required in many commercial operations, such as the manufacture of acetylene, sulphuric acid, bleaching-powder, saltpetre, and the nitric acid esters (nitro-glycerin, gun-cotton, &c.).

At the end of the volume is a number of tables useful in gas analysis.

The book is well arranged, and the descriptions of the various apparatus are clear and well illustrated; there is a good table of contents and an index.

Subject List of Works on Domestic Economy, Foods, and Beverages, in the Library of the Patent Office. London: The Patent Office. 1902. Pp. 136.

This list, which comprises works on the culture of cocoa, coffee, barley, hops, sugar, tea, and the grape, is arranged in a manner similar to those which have already been noticed in this column (CHEMICAL NEWS, vol. lxxv., pp. 23 and 238). It includes a general alphabet of subject headings, with descriptive entries, in chronological order, of the works arranged under these headings; and a key or summary of these headings shown in class order. The present list comprises 1270 works, representing some 2043 volumes. The catalogue entries relating to these works number 1592, and are distributed under 174 headings and sub-headings.

Aids to Practical Dispensing. By C. J. S. THOMPSON, M. Pharm. Soc., Third Edition, Enlarged, with Illustrations. London and Dublin: Baillière, Tindall, and Cox. 1902. Pp. 92.

THE object of this work is mainly to lay before those students who have not had the opportunity of much practice, the various methods employed in dispensing medical prescriptions, and to enable them to become familiar with the necessary processes.

Special attention has been devoted to the more difficult operations, and practical hints are given in the making of emulsions, suppositories, and plasters.

The appendix contains, among other things, a list of the Latin terms used in prescriptions, with their English meanings, as well as a useful table of solubilities. There is a table of contents, and an index.

The Evolution of Artificial Mineral Waters. By WILLIAM KIRKBY, F.L.S., Lecturer on Pharmacognosy in the Owens College. Manchester: Jewsbury and Brown. 1902. Pp. 155.

THE object with which this book was written was to furnish a connected account of the initiation and development of the mineral water industry, which has achieved a more satisfactory position in England than in other countries, with the exception of America. Detailed technical descriptions of machines and processes have been avoided as much as possible, as the book is not intended as a manufacturer's handbook.

The first successful attempt to impregnate water with carbonic acid gas appears to have been made by Brownrigg in the year 1741, and later on Dr. Joseph Priestley devised a method for the more convenient use of this "new medicinal agent."

The application of the compression pump became quite common early in the nineteenth century, and in this country Bramah's machine has been found to be the only one giving a really continuous supply, though other makers have attempted to give a personal impress to their productions by adopting different designs.

An interesting development of the syphon is an adaptation which has made it possible to aerate the contents of the syphon directly by means of bulbs of condensed carbonic acid gas. These reservoirs are now generally known as "sparklets," and are looked upon as quite a new invention, but it is astonishing to read that they were invented and fully described so far back as 1874 by Arthur Marescot.

The book is profusely illustrated, and is provided with a good index.

In the copy before us there is a curious mistake; the frontispiece, "Priestley Exhibiting his Apparatus to the Fellows of the College of Physicians," has printed over it the medallion portrait of "Dr. Priestley, F.R.S.," which also faces p. 61.

Report of the Senior Analyst at the Cape of Good Hope, for the Year 1901. Cape Town: W. A. Richards and Sons. 1902. Pp. 61. With two Maps.

THE continuance of the war during the year 1901 brought about a temporary suspension of two important branches of the work of this department. The systematic investigation into the composition of the soils of the colony was entirely stopped, and the operation of the Adulteration Act has also been all but entirely suspended; however, in spite of these set-backs, the number of samples analysed during 1901 was only 559 less than in 1900; so that there is every indication that in branches not affected by the unsettled state of the country, the onward movement is as steady as ever. The actual number of samples analysed during the past four years is as follows:—

1898..	..	..	..	..	..	1650	samples
1899..	..	..	..	..	..	1903	"
1900..	..	..	..	..	..	1949	"
1901..	..	..	..	..	..	1389	"

Of these, the largest number of analyses during 1901 were of milk, viz., 566 samples; water, soils, fertilisers, tinned-meats, and ink are among the other materials which claimed most attention.

Beginners' Guide to Photography. Eighth Edition, Revised and Enlarged. London: Messrs. Perkin, Son, and Co., Ltd.

THE enterprising publishers of this "Guide" are to be congratulated for having collected together in a convenient form a mass of useful photographic information. At the present day when almost every schoolboy is a photographic aspirant, a little genuine instruction that will give an intelligent idea of the reasons for performing the various operations involved in making a photographic picture is of real value. Details of dry-plates, development, the chemicals used, stereoscopic photography, lantern-slide making, enlargement, &c., are given clearly and in a thoroughly practical manner. Unfortunately, there is neither a table of contents nor any index. This is a great pity, and detracts much from the usefulness of the book. There are 226 pages, of which 89 are devoted to advertisements, and we are glad to see that the latter are all together at the end and not, as in the case of some books, interleaved with the instructions.

Guide to Preparation Work in Inorganic Chemistry; for Students of Chemistry and Pharmacy. By Dr. REINHART BLOCHMANN, Professor at the University of Königsberg. Authorised Translation, by JAS. LEWIS HOWE, Washington and Lee University.

THIS book, written in the strictest fashion of American spelling, is intended to serve as a guide to students in inorganic preparation work. That the rational preparation of chemical compounds must always rest upon the equivalent proportions of the reacting substances is insisted upon; the time necessary for the various operations is indicated, and in cases where by-products are

formed the recovery of these in a useful form is carried out. The methods for the preparation of some twenty-five compounds are given with clear illustrations of the forms and disposition of the apparatus needed in each operation; while at the end of the book are tables of atomic weights, specific gravity, and solubility.

The book is issued by the Department of Chemistry, Washington and Lee University, Lexington, Virginia.

OBITUARY.

PROFESSOR HENRY MORTON, Ph.D.

We regret to announce the death of Dr. Henry Morton, which occurred recently at Hoboken, New Jersey, U.S.A.

Dr. Morton has been well known for many years in connection with the Stevens Institute of Technology, Hoboken, of which he was the President from its foundation in 1871, in accordance with the will of the late Edwin A. Stevens.

Dr. Morton was the author of many scientific papers, his first one being on "An Important Adjunct to the Induction Coil," published in the *CHEMICAL NEWS* in 1867; in the following year he published two more papers, viz., "Sunlight and Moonlight," and "Resistance and Transmission of Motion," followed by the "Solar Eclipse of August 7th" in 1866.

He was the author of several papers on colour and fluorescence, and in conjunction with Prof. Carrington Bolton did some important and interesting work on "The Fluorescence and Absorption Spectra of the Uranium Salts" in 1874; another highly fluorescent body called "Thallene" was the subject of a paper published in 1876, in which Dr. Morton gave a description of its source and the history of its discovery.

Optical subjects, however, were not the only ones dealt with by Prof. Morton; in 1878 he wrote a paper on "The Singing Telephone at the Stevens Institute," and another "On the Elimination of Antimony from the Human Organism," while in 1879, in conjunction with Mr. W. Geyer, he published a paper entitled "Experiments illustrating the Formation of 'Fast-red or Rocceline,'" and another in 1880 on "A New Sulpho-Acid of Phenanthrene."

Though Prof. Morton's publications ceased in 1880, he by no means retired from active life at that date, but devoted his whole energies to the welfare of the Institute of which he has been so many years the distinguished head.

A Compound of Bromide of Aluminium with Bromine and Sulphide of Carbon.—V. A. Plotnikof.—If, to a solution of bromide of aluminium in sulphide of carbon, we add gradually a little bromine or a solution of bromine in sulphide of carbon, an oily liquor is formed at first, of a dark red colour, which, as the quantity of bromine added increases, becomes transformed into a yellowish-red solid body. When washed with pure sulphide of carbon and dried *in vacuo*, this body is in the form of a yellow amorphous powder, appearing greenish by reflected light; it rapidly becomes red when exposed to direct sunlight; a trace of moisture produces the same effect. It will remain for a long time without change when kept in sealed tubes in the dark; it melts at 86–90° with decomposition, leaving bromide of aluminium, $AlBr_3$; it is soluble in ether, sulphide of carbon, bromide of ethyl, bromide of ethylene, and benzene. Analysis indicates the composition $AlBr_3 \cdot Br_2 \cdot CS_2$. Water decomposes this body with the evolution of heat and the formation of a large quantity of gas; a deep red-coloured oil is produced which is easily transformed into the crystallised trithio-bromide, $C_2S_3Br_6$.—*Journ. Soc. Phys. Chim. R.*, vol. xxxiii., p. 91.

CORRESPONDENCE.

PROPOSED ELECTRO-CHEMICAL SOCIETY.

To the Editor of the *Chemical News*.

SIR,—I notice in the *CHEMICAL NEWS* for October 3rd (vol. lxxxvi., p. 174), under the heading of "Notes and Queries," a query from Dr. Max Reuter enquiring whether there is a Society of Electro-chemists in London. Readers of the *CHEMICAL NEWS* may perhaps be interested to learn that there is a proposal on foot to form an Electro-chemical Society in this country. To this end, a small committee has been holding occasional meetings since March, and has recently sent out circulars to various chemists and others interested in electro-chemical science. A very fair response has been received; but I should like to point out, through the medium of the *CHEMICAL NEWS*, that the response has been mainly from those interested in the purely electrical side of the subject; chemists being inclined to hang back, in order, apparently, to see whether the Society can be formed before they will lend a helping hand. At the same time several prominent chemists have promised their support.

It is felt by those interested in the formation of the Society that electro-chemistry is not sufficiently known and applied in this country, and it is hoped that if an Electro-chemical Society is formed interest may be awakened, and both science and industry be the gainers. Germany and America have electro-chemical societies, France and Russia are experimenting and founding large industries. Our electricians desire to introduce new processes, but are largely handicapped by want of chemical knowledge; they want help from the chemists. The Society, if formed, should bring the chemists and electricians together, and both would benefit by the interchange of ideas.

I shall be very pleased to send particulars of the proposed Society to any chemists who may be interested in the subject; or they can obtain information from Mr. S. V. Simpson, Grosvenor Mansions, Victoria Street, who is Hon. Secretary to the Committee.—I am, &c.,

F. MOLLWO PERKIN.

Borough Polytechnic Institute, S.E.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 12, September 22, 1902.

Compounds of Silicon with Cobalt, and a New Silicide of the Metal.—P. Lebeau.—The analogy existing between the formulae and properties of the two known silicides of cobalt and those of the silicides of iron, led the author to suspect the existence of a third compound, richer in silicon and comparable with Si_2Fe . His researches confirmed this prediction. Such a compound can be produced by heating cobalt in presence of an excess of molten silicon, or by the action of the temperature of the electric furnace on a mixture of copper silicide, cobalt, and silicon. In the latter case, the compound is better crystallised and more easily purified. Analyses made on various specimens of this substance show that it has the formula Si_2Co . It always contains, however, as an impurity, a little carbon silicide. It is found in the form of little dark coloured crystals, of density 5.3.

The Calorific Power of Oil.—M. Goutal.—The author endeavours to establish a relation between the calorific power of coal and that of combustibles which are

commonly used, by the process of calcination, incineration, and desiccation, in order to determine the fixed carbon, the volatile materials, the ash, and the humidity. His experiments show that the calorific power of pure anthracite has the mean value of 8250 cal., that of anthraciteous oils 8550 cal. A maximum of 8700 cal. is attained for coal where the volatile matter is from 10 to 30 per cent of the whole. The calorific power of oils increases in proportion as their volatile matter decreases, up to the limiting value of 30 per cent, after which the calorific power of natural combustible materials and that of their volatile matter diminishes concurrently.

MISCELLANEOUS.

"Ore in Sight."—As a result of the discussion of a paper recently read before the Institute of Mining and Metallurgy by Mr. J. D. Kendall, the Council of the Institute recommend, among other things, that members should not make use of the term "Ore in Sight" in their reports, without indicating, in the most explicit manner, the data upon which the estimate is based; the term in question is frequently used to indicate both "ore blocked out" and "ore which may be reasonably assumed to exist" though not actually blocked out. In using the term "ore in sight" an engineer should demonstrate that ore so described is capable of being profitably extracted under the working conditions obtaining in the neighbourhood. May we suggest that the term "in sight" is probably a corruption of "*in situ*," which has a very different meaning, and would naturally be liable to misconception if used indiscriminately.

German Apparatus in New Zealand.—We have received from the Agent-General for New Zealand a cutting from *The Lyttleton Times*, in which Dr. Evans, of Canterbury College, gives a list of nine reasons why the chemical apparatus for use in the college should be obtained from German firms instead of English. These nine reasons form a scathing criticism of the business methods of English firms. Some of the reasons are as follows:—Many of the lines required are not made in England at all; the German goods are, as a rule, made of better material, and are very much better finished; the prices charged by most English dealers are exorbitant, and in no way represent the actual value of the goods supplied; the goods, when ordered from an English firm, very often prove to be of French or German make, but in addition the goods are not seldom seriously damaged by deliberate "tinkering" on the part of the English dealer; if articles are ordered "as per plan enclosed," the overseer of the German house will see that his workmen do work according to the plan, while many an English firm rests content if, after many days, it returns you something "distinctly resembling" your plan. Dr. Evans goes on to point out that it would be distinctly unfair to make such a series of charges without supporting them, where possible, with definite instances; this he proceeds to do in detail, quoting at first only instances which have come under his own personal observation, and confining himself, in the main, to the experience of the last three years only. In conclusion, Dr. Evans writes that it is but fair to state that the above criticisms, as a whole, do not apply to English electrical apparatus of the better class, nor is it difficult to get first-class work done, at a somewhat higher price it is true, in the optical and acoustical branches.

Decomposition of Hypsulphite of Sodium by Acids.—H. V. Cöttingen.—It is well known that the addition of an acid to a solution of hypsulphite of sodium only causes a precipitate after the lapse of some instants. The author has devised an apparatus by which he can accurately determine the time which elapses between the mixing of the two liquids and the appearance of the precipitate; by this means he has been able to

ascertain that the time, y , depends on the concentration x , of the hydrogen ions in the acid liquor employed, the law of this dependence being represented by the equation—

$$y = \frac{1}{A \log (1 + bx)}$$

where A and b represent two constants. The time is therefore the same for isohydric acid solutions. The addition of a solution of sulphite of sodium retards the formation of the precipitate of sulphur; the relation between x and y keeps the same logarithmic form, but the values of the constants A and b depend on the quantity and the concentration of the sulphite added. The reactions observed by Colefax (*Chem. Soc.*, 1892, vol. lxi., p. 176 and 199) between hyposulphite and sulphite of sodium did not permit of the study of the state of equilibrium to which the reaction—

Hypsulphite = sulphite + sulphur

may give rise. Finally, the influence of the concentration of the ions of hydrogen in the acid liquor makes itself felt, not only on the time which elapses before the appearance of the first cloudiness, but also on the speed with which the precipitate continues to be formed; measurements of conductivity have, in fact, enabled it to be observed that this speed is the same in the cases of isohydric acid solutions.—*Zeit. Phys. Ch.*, vol. xxxiii., p. 1.

Electrolysis of the Alkaline Salts of the Organic Acids.—J. Petersen.—The electrolysis was done at 0° on slightly acid solutions of the potash salts of the acids under examination. Under these conditions, formate of potassium gave approximately equal volumes of hydrogen and carbonic acid (the result of oxidation at the anode), with a small quantity of oxygen. In dilute solutions the amount of carbonic acid obtained increases with the density of the current; in concentrated solutions the disengagement of oxygen at the anode almost ceases. Acetate of potassium gives ethane and hydrogen in proportions which vary with the strength of the current; carbonic acid gas is also given off at the anode; the formation of ethylene has not been observed, neither is there any acetate of methyl formed. Propionate of potassium gives butane, propionate of ethyl, ethylene, and carbonic acid gas at the anode, while hydrogen is given off at the cathode. Butyrate of potassium gives isopropyl alcohol, butyrate of isopropyl, hexane, and apparently butyrate of propyl; at the same time, the gaseous mixture obtained contains 1 volume of propylene for each 2 volumes of hydrogen. Finally, isobutyrate of potassium gives isobutyrate of isopropyl, isopropyl alcohol, and a little di-isopropyl, and, at the same time, hydrogen and propylene.—*Zeit. Phys. Chim.*, vol. xxxiii., p. 99.

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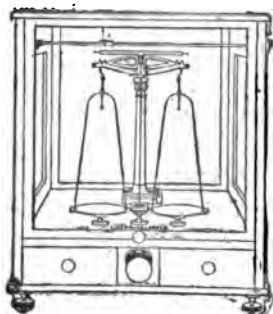
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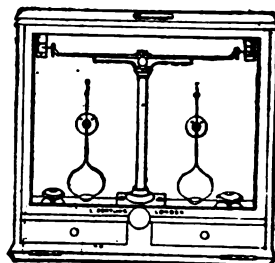


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THE RADIO-ACTIVITY OF URANIUM.

By FREDERICK SODDY.

DURING the course of the investigation contained in the preceding paper on the radio-activity of thorium, it became advisable to investigate in parallel a radio-active element that does not give out an emanation, or excite radio-activity upon surrounding bodies. It has been shown that these secondary actions always form a possible factor in the results obtained with thorium. For this purpose the radio-activity of uranium was chosen, but at the outset great differences arose between the results obtained and those published in the same field by prior investigators. These differences led to a comparative study being made in certain cases of the photographic and electrometer methods of measuring uranium radio-activity, which resulted in an explanation of the anomalous results that had been obtained.

Sir William Crookes (*Proc. Roy. Soc.*, 1900, p. 409) made the beautiful discovery that in one chemical operation uranium could be obtained inactive to the photographic plate, while the whole of the inactivity was concentrated in a small non-uranium residue. This residue, to which he gave the name $U'X$, was at least a hundred times more active than the uranium from which it had been separated.

On repeating this separation, which consists in precipitating solutions of uranium with ammonium carbonate, and re-dissolving the precipitate in excess of the reagent, it was found that the small residue of $U'X$ was almost inactive, while the uranium, contained in the ammonium carbonate filtrate, possessed about the normal amount of activity. The electrical methods of Professor Rutherford were employed for measuring radio-activity. The same negative result was obtained by the other method of separation employed by Crookes. If crystallised uranium nitrate is dissolved in ether, the uranium divides itself in two unequal fractions between the ether and water present. That dissolved in the ether layer, which is the major fraction, was found by Crookes to be inactive to the photographic plate, while the small part dissolved in the aqueous layer possessed all the activity contained in the original uranium nitrate. But as in the former case, on repeating the operations, both fractions appeared almost equally active to the electrometer, and no separation had apparently been effected.

The same preparations were then examined for their action on the photographic film, and gave results exactly the converse of those observed with the electrometer and in accord with the published results of Sir William Crookes.

Rutherford (*Phil. Mag.*, 1899, p. 109) showed that uranium radiation consists of two different types, which he named the α and β radiation. The α is absorbed very readily even by gases, being reduced to half value by passage through 4.3 m.m. of air. The β , on the other hand, is very penetrating in character, and is but little absorbed by gases. It is able to pass through 0.5 m.m. of aluminium before it is reduced to half value. This latter part constitutes only a few per cent of the total radiation of the uranium. In addition, by the electrometer method of measurement under ordinary circumstances, a fraction only of the intensity of the β radiation is recorded, for this method depends upon the ionisation of the air, and this is proportional to the absorption of the rays by the air. It follows, therefore, that under ordinary circumstances, in which the rays traverse a few centimetres only of air,

that the electrometer method measures practically the α radiation of uranium alone. The photographic method employed by Crookes, in which the rays are made to pass through glass or card before reaching the sensitive film, will, on the other hand, measure only the β radiation. Hence the results obtained would be explained if the methods employed separated the β radiation only, and left the α radiation intact.

Bequerel has shown (*Comptes Rendus*, 1900, cxxx., p. 1584) that the rays from uranium are deflected in the magnetic field, and therefore consist of cathode rays. Rutherford and Grier (*Phys. Zeit.*, 1902), in a general investigation of the rays from various radio-active substances to determine what proportion in each case was deviable in the magnetic field, and therefore cathodic in nature, had already at this time shown that the β radiation of uranium is entirely cathodic, and the α radiation not at all. They undertook the examination of the specimens that had been prepared in the course of the present work, the details of which appear in their paper with the special apparatus employed. It suffices to state here that in the arrangement they adopted the rays are made to pass through a sufficient thickness of air to absorb a considerable part, and the sensitive Dolezalek electrometer was employed. Under these circumstances $U'X$ was found to be giving out ionising rays abundantly. This radiation was shown to be the β radiation of uranium only, practically free from α , for it passed through aluminium foil without great loss, and is deviable to the same extent by the magnetic field. The rays from the photographically inactive uranium was found, on the other hand, to consist entirely of a radiation, and gave no rays deviable in the magnetic field, or capable of passing through thin aluminium foil. I must here express my great obligation to Professor Rutherford and Mr. Grier for permission to use these results.

It thus appears that the methods of chemical separation employed by Crookes effected a separation only of the constituent responsible for the β radiation. The uranium after the process still retains the whole of the non-deviable radiation, which in the electrometer method contributes the greater part of the effect. In this respect uranium is analogous to thorium, as shown in the preceding paper. But the excited radio-activity produced by ThX as a secondary effect, which itself comprises both deviable and non-deviable radiation, makes it impossible at present to say whether the primary radiation of ThX is, like that of $U'X$, wholly cathodic or not. The non-separable parts in both cases consist entirely of rays non-deviable in the magnetic field.

The next point to be decided was whether the α radiation of uranium could affect the photographic plate. A sample of the preparation free from β radiation was exposed about 5 m.m. away from a sensitive film for seventy-two hours without intervening screen. Only a very slight darkening occurred, so that the action, if any, of the α radiation may be considered negligible in comparison to the effect of the β radiation. It is not possible to try longer exposures than three days by this method, owing to the regeneration of the β radiation which will be referred to later. However, Becquerel (*Comptes Rendus*, 1902, cxxxiv., p. 208) exposed plates for twenty and forty-nine days near uncovered uranium salts, in a magnetic field where the β radiation would be deflected and eliminated, and failed even after these long periods to obtain an impression on the sensitive film. He concluded that the whole of the uranium radiation is cathodic, and can be deflected by the magnetic field. But the foregoing considerations make it clear that the α or non-deviable radiation of uranium, which contributes the major part of the ionisation effect, is without appreciable action on the photographic plate.

In light of these results, and those obtained for thorium in the preceding paper, the method employed by Becquerel (*Comptes Rendus*, 1900, cxxxi., p. 137) to separate the active constituent of uranium is of interest. M. Becquerel

precipitated barium as sulphate in solutions of uranium, and found that after successive precipitation the activity of the latter was much enfeebled, both towards the electrometer and the sensitive film. As, however, he enclosed the salt in paper for the electrometer measurements, and this absorbs the α radiation almost or quite completely, the result must not be taken to mean that both the α and the β radiations were separated by the process. On repeating the separation it was found that, as in the case of the methods of Crookes, the β radiation only was removed. After four successive precipitations in one day, the β radiation was found to be reduced to 8 per cent of its original value, and eight more precipitations on the day following completely removed it. But the activity of the resulting uranium preparation to the electrometer under ordinary circumstances was hardly appreciably decreased. The α radiation had therefore not been affected.

Bequerel has already shown (*Comptes Rendus*, 1901, cxxxiii., p. 977) that the uranium after this treatment recovered its normal activity on standing. With the sensitive electrometer the presence of cathode rays in a product originally quite freed from them, can be detected after three days. It thus appears probable that what has been shown to hold true for ThX applies equally to UrX, and the subject is under investigation conjointly with Professor Rutherford. The same question therefore arises for the non-separable activity of uranium already discussed for that of thorium.

1. Is this residual activity to be regarded as a secondary radiation produced by the presence of UrX?
2. Or, is it caused by a distinct material substance capable of chemical separation?

On the first view, if UrX—the cause of the phenomenon—is removed, the residual activity will decay with time. The following experiment was therefore performed:—The solution of uranium nitrate which had been successively precipitated with barium sulphate twelve times, and had been shown to be free from β radiation, was allowed to stand. Every three or four days it was once more precipitated in the same manner, and the UrX produced during that period removed from the uranium. After twenty-one days, the uranium in the solution was precipitated with ammonia and converted into the green oxide. For comparison, a sample of the same uranium nitrate, as obtained from the maker, was subjected to two precipitations with barium sulphate to remove most of the β radiation, the uranium then being precipitated and converted into oxide as in the former case. The radiations from the two samples were then directly compared. In the one the α radiation had been given three weeks to decay, and by comparison with the other a direct measure of the diminution suffered during this period could be obtained. Not the least difference, however, could be detected in the intensities of the radiations in the two cases. The actual values obtained were within 1 per cent of each other. If it is assumed that in this experiment a difference of 5 per cent could be with certainty detected, the conclusion is arrived at that if the α radiation of uranium is a secondary phenomenon produced by the β , it takes at least a year to decay to half value. As this is not in accordance with what is known of the nature of excited radio-activity, the view cannot be regarded as probable.

On the second view, the α radiation is produced by a second distinct type of matter. But as in the case of thorium, all attempts made to separate such a constituent of uranium by chemical methods have so far failed, although the methods have not yet been exhausted. It is, however, interesting to note that polonium, discovered by Curie, fulfils in almost all respects the functions of this hypothetical constituent. It gives only non-deviable radiation, and Professor Rutherford to whom the suggestion is due, found that the penetration power of the rays from polonium is very similar to that of the uranium radiation. Moreover, M. and Mme. Curie (*Comptes Rendus*, 1902, cxxxiv., p. 85) have stated that its

activity slowly decays with time, which is to be expected after, but not before, it has been separated from the uranium producing it. However, experimental work has not yet justified this suggestion.

The actinium of Debierne, according to a recent paper by Crookes (*CHEMICAL NEWS*, lxxxv., p. 109), has been found to be identical with UrX, although no reference is given.

In the paper just quoted, Crookes brings forward some results obtained by the photographic method which led him to the conclusion that UrX gives a radio-active emanation similar to that given by thorium and radium. No evidence of such a radio-active emanation has been obtained in the course of the present work even with the most sensitive electrometer, and it appears probable that the cause of the darkening of the photographic plate by this body in the cases where the film is shielded from direct radiation, is not a radio-active substance in the accepted sense of the word, but an agent similar to hydrogen peroxide in its photographic actions.

Macdonald Chemistry and Physics Buildings,
McGill University.

ON PERCHLORIC AND PERIODIC ACIDS.

By A. ASTRUC and H. MURCO.

THE thermochemical examination of perchloric and periodic acids has shown that singular differences exist between these two bodies. Thus, perchloric acid behaves as a strong acid, decidedly monobasic, giving off 14.25 cal. with soda, with complete solution; an excess of base or acid has no sensible influence on the neutral salt, and the perchlorates are very stable.

Periodic acid behaves differently; according to the amount of potash used for its neutralisation it gives off variable quantities of heat, so that MM. Basarow, Thomsen, and Berthelot all regarded it as a bibasic acid, with a manifest tendency to form not only acid salts, but also basic salts containing up to 5 atoms of a monovalent metal.

Ferlund looked upon periodic acid as being tribasic, Rammelsberg tetrabasic, while Langlois and Leutsch thought it was pentabasic.

The comparative examination of the action exercised by these two acids on some of the colouring reagents generally used in acidimetry, gave us results of a similar kind.

Our experiments were carried out on the hydrate of perchloric acid, $\text{ClO}_4\text{H}_2\text{H}_2\text{O}$, and the hydrate of periodic acid, $\text{IO}_4\text{H}_2\text{H}_2\text{O}$, the composition of both of which was previously verified by analysis.

With the following indicators, viz., heliantine A, phenolphthalein, Poirrier's blue, tincture of litmus, and rosolic acid, the hydrate of perchloric acid behaved as an exactly monobasic acid; the reactions were quite sharp.

The hydrate of periodic acid behaved altogether differently. In the presence of heliantine A it is saturated by one molecule of potash, soda, or ammonia, and by half a molecule of lime, baryta, and strontia; the reactions are sharp and very distinct.

The results obtained are very different if we use phenolphthalein; in this case the reactions are progressive, generally uncertain, and vary with the alkaline solution used. The rose colouration of the reagent appears after the addition to one molecule of periodic acid, of 1.5 to 1.7 molecules of potash, soda, or ammonia; under the same conditions, two molecules of acid require 1.9 to 2.0 molecules of baryta and 2 to 2.3 molecules of strontia and lime.

Litmus and rosolic acid both give results similar to the above, but the reaction is still less distinct; Poirrier's blue gives results practically agreeing with those obtained with phenolphthalein.

The thermochemical observations are thus confirmed

by these alkalimetric experiments. Perchloric acid is a strong acid comparable with hydrochloric acid, nitric acid, &c. Periodic acid is a complex acid possessing various acid functions, one which acts on heliantine A being strong, and the others weaker and influencing the other indicators.

A further result of these experiments is that all these colouring agents can be used for the titration of perchloric acid, and only heliantine A should be used with periodic acid.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 18.

THE COMPLEX SALTS OF OSMIUM. OSMYLOXALATE OF POTASSIUM.

By M. VÉZES and L. WINTREBERT.

I. Action of Oxalic Acid on a Potassic Solution of Peroxide of Osmium.—If crystallised oxalic acid is added gradually to a solution of peroxide of osmium in potash-lye, keeping the mixture at boiling-point in a flask fitted with a vertical condenser, the following phenomena are successively noticed:—First of all, the potash and the peroxide being in excess with regard to the oxalic acid, we observe that the mixture, which is coloured a deep brownish red at first, takes the reddish violet tint characteristic of solutions of osmiate of potash, OsO_4K_2 , and at the same time carbonic acid is given off; the oxalate of potassium formed in the solution thus plays the part of a reducing agent under these conditions, and acts on the peroxide of osmium in the same manner as alcohol or nitrite of potassium in the classic methods of preparing osmiate of potassium, described for the first time by M. Fremy (*Ann. Chim. Phys.*, 1844, Series 3, vol. xii., p. 516). The reduction which takes place in the solution can be represented by the equation:—



If we increase the amount of oxalic acid used, we observe the appearance of a black pulverulent precipitate in the body of the violet liquor; this is, according to all appearance, the osmic acid, OsO_4H_2 , described by Morah and Wischia (*Zeit. Anorg. Ch.*, vol. iii., p. 156). This body, which is always formed when we have an osmiate in acid solution, takes its rise in this case from the presence of a slight excess of oxalic acid over the quantity corresponding to equation 1.

The addition of a further quantity of oxalic acid has the effect of re-dissolving this precipitate and giving an opaque solution of a blackish brown colour, which finally, if kept at the boiling-point with the addition of a sufficient excess of oxalic acid to give the solution a distinctly acid reaction, becomes almost decolourised, forming a bright yellowish-brown solution.

On cooling, this solution deposits brown crystals, having under the microscope the appearance of fine birefrangible prismatic needles. As we shall see later on, the composition of these crystals can be represented by the formula $\text{OsO}_2(\text{C}_2\text{O}_4)_2\text{K}_2 \cdot 2\text{H}_2\text{O}$, and we have given them the name of osmyloxalate of potassium. This salt is the result of the following reaction between the osmiate (or osmic acid) first formed, and the excess of oxalic acid used (or its monopotassic salt):—



Disregarding the intermediate products, the following equation, obtained from equations 1 and 2, expresses the formation of the same salt from the primary materials first put into reaction—peroxide of osmium and potash:—



Of the two reactions 1 and 2, which go to form osmyloxalate of potassium, the second takes place very rapidly; we shall see later on that a solution of osmiate of potas-

sium, treated with an excess of oxalic acid, gives almost immediately an abundant deposit of osmyloxalate. On the other hand, the first of these reactions, the reduction of the peroxide, only takes place slowly and after prolonged boiling. If, in fact, to a given quantity of peroxide dissolved in the equivalent amount of potash, we add, all at once, an excess of oxalic acid (a little more than the three molecules required by equation 3), we obtain almost immediately the final decolouration of the liquor, and this latter on cooling deposits the osmyloxalate; however, it only gives a small quantity, and at the same time it deposits, first in the melted state and then solid, the greater portion of the peroxide used. If the whole is now re-heated, the osmyloxalate and the peroxide both re-dissolve; after boiling for an hour, a fresh cooling will give much more osmyloxalate and much less peroxide than in the former case. However, the complete disappearance of the peroxide and its integral transformation into osmyloxalate cannot be effected except after further prolonged boiling for several hours.

II. Preparation of Osmyloxalate of Potassium.—In consequence of the above observations, the following method should be used for the preparation of osmyloxalate of potassium:—

A known weight of peroxide of osmium (say, about 20 grms.) is placed in a two-litre flask with an aqueous solution of pure potash (15 grms. in 500 c.c. of distilled water) and an excess of crystallised oxalic acid (50 grms.), so that the potash, and especially the oxalic acid, may be in excess with regard to the relative quantities required by equation 3. The neck of the flask must be connected with a vertical condenser in such a manner as to prevent as far as possible any contact of the fumes of the peroxide with any organic substance; grinding with emery powder will do very well, or an indiarubber ligature round two tubes of slightly different diameters, one fitting well into the other; these being the condenser tube and the neck of the flask. Finally, at the upper end of the condenser a bulb-tube is attached in a similar manner; this tube contains a little potash solution to retain any fumes of the peroxide that might otherwise escape from the condenser.

The apparatus being thus fitted up, the contents of the flask are brought to boiling-point; besides carbonic acid, fumes of peroxide are given off which condense in the condenser and fall back into the flask in the fused state. The boiling is continued until the evolution of peroxide fumes ceases; this requires several hours (at least three hours with the quantities given above). During this time, the contents of the bulb-tube must be frequently renewed as the potash is transformed into carbonate by the carbonic acid given off, and therefore becomes less able to arrest the osmic fumes; the partial decolouration that takes place, passing from deep reddish brown to bright yellow, shows the time when this renewal should be made.

When no more fumes of peroxide condense, the boiling is stopped; further boiling would interfere with the reaction, especially in the presence of a large excess of oxalic acid, since products of reduction of the osmyloxalate would be formed.

The limpid solution in the flask is allowed to cool, and soon a considerable deposit of crystals of osmyloxalate of potassium appears. After complete cooling, the whole of the contents of the flask is decanted into a Büchner funnel, in which the crystalline deposit is rapidly freed from the mother-liquor by means of the filter-pump. It is then further dried by squeezing between filter-papers.

When carried out with the above-mentioned precautions, the operation is not dangerous, as the crystals can be collected without any inconvenience from the fumes of the peroxide. Further, the return is almost quantitative (38 grms. for 20 grms. of peroxide); as a rule, the quantity of the salt corresponds with the fumes of peroxide collected in the potash tubes, and this latter can be used in subsequent operations.

Finally, the osmyloxalate of potassium, being very slightly soluble in the cold, as well in solutions of the

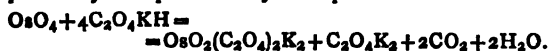
			Calculated.		Found.								
					I.	II.	III.	IV.	V.	VI.	VII.	Mean.	
Os	..	..	191.0	37.21	—	37.59	37.46	37.55	—	37.34	37.11	37.41	
2K	..	..	78.3	15.25	—	15.06	15.27	15.51	—	15.24	15.10	15.24	
4C	..	..	48.0	9.35	—	—	—	—	9.53	—	—	9.53	
10O	..	..	160.0	31.17	—	—	—	By difference		—	—	30.78	
2H ₂ O	..	..	36.0	7.02	6.93	—	7.19	7.02	—	7.03	—	7.04	
OsO ₂ (C ₂ O ₄) ₂ .K ₂ .2H ₂ O			513.3	100.00								100.00	

ordinary alkaline salts as in water, it is easily obtained in the presence of these salts; consequently, its preparation constitutes an excellent means for recovering, in the form of a stable and well-defined salt, the osmium from residues in which it is contained, so long as the metal is given off in the form of peroxide, although at the same time mixed with various acid fumes.

III. *Other Methods of Preparation.*—Besides the fundamental method of preparation which has just been described, two other methods may be used for obtaining osmyloxalate of potassium.

The first, the existence of which confirms one of the essential points of the theory already put forward, consists in the direct production of the second of the two reactions by which we explained the formation of the osmyloxalate from the peroxide. It suffices to make a concentrated solution of osmate of potassium, and to treat it while hot with an excess of oxalic acid, to obtain immediately, through the cooling of the mixture, an abundant deposit of osmyloxalate according to equation 2. The slight stability of aqueous solutions of osmate of potassium, which, especially when hot, become strongly impregnated with a black deposit of osmic acid, forms no obstacle to this operation; the deposit is re-dissolved, as has been seen above, in an excess of oxalic acid; however, these solutions can be made relatively stable by the addition of a small quantity of potash. In such a case the black precipitate of osmic acid will only appear transiently during the course of the reaction, when the oxalic acid, added to the solution in the crystalline form, is still only partially dissolved.

We can also obtain the same salt by digesting peroxide of osmium with a concentrated solution of binoxalate of potassium. The mixture being made in the cold, and left in a ground stoppered flask, no reaction is observed at first, but after some months the solid peroxide disappears, and we find beautiful crystals of osmyloxalate of potassium at the bottom of the flask. The reaction which takes place may be represented by the equation:—



Though very slow in the cold, the reaction is accelerated by raising the temperature, but in the latter case more than in the former it is necessary to loosen the stopper occasionally to prevent the pressure of the carbonic acid, which is continually coming off, from becoming excessive. We may add that this method, though less practical than the preceding ones if quantities of osmyloxalate are required quickly, is, on the other hand, the only one by which crystals can be obtained sufficiently large for purposes of measurement with the goniometer.

IV. *Composition.*—The analysis of osmyloxalate of potassium offers special difficulties which do not occur in the case of the complex oxalates of the other metals of the platinum group; the ease with which finely divided osmium is oxidised on contact with the least trace of free oxygen, giving fumes of peroxide, necessitates the whole of the analytical operations being carried out in a current of hydrogen perfectly free from air.

As the method of procedure we adopted will be described shortly by one of us, we will restrict ourselves here to the results obtained.

The analyses I., II., III., IV., and V. were carried out on samples prepared by means of peroxide of osmium, oxalic acid, and potash; analysis VI. on a salt obtained

from the osmate, and number VII. on a salt prepared from peroxide and binoxalate of potassium.

Quite independently of proofs of a different kind which will be given later on, the analysis suffices to show that osmyloxalate of potassium is a hexavalent derivative of osmium, an osmyl salt comparable with osmylsulphite of sodium, $\text{OsO}_2(\text{SO}_3\text{Na})_2$, recently described by Rosenheim and Sasserath (*Zeit. Anorg. Ch.*, vol. xxi., p. 124), and to the salts of osmyldiammonium, more especially the chloride, $\text{OsO}_2(\text{NH}_3)_2\text{Cl}_2$, the composition of which was elucidated in 1881 by Wolcott Gibbs (*Amer. Chem. Journ.*, vol. iii., p. 223).

V. *Properties.*—The crystallographic examination of osmyloxalate of potassium, which we owe to the kindness of H. Dufet, gave the following results:—

Triclinical crystals, formed of the facets, ρ (001), $f\frac{1}{2}$ (111); dominants, h_1 (100), g_1 (010), $d\frac{1}{2}$ (111), $b\frac{1}{2}$ (111), x (131), y (131).

The three latter are rare and small; the facets ρ and $d\frac{1}{2}$ are often imperfect.

Parameters .. $a:b:c::0.50044:1:1.05503$.

The optical sign is positive. One of the optical axes practically coincides with the normal to ρ (001). The plan of the axes makes an angle of 47° with the edge $\rho f\frac{1}{2}$ (001) (111), and of 77° with the edge ρg_1 (001) (010). The angle of the axes, observed in air on the face ρ , is $64\frac{1}{2}^\circ$, the axis inclined to the normal is at the side of the angle ρg_1 (111) (010).

Dichroism is noticeable; in ρ the colour is greenish yellow for vibrations parallel to the plan of the axes, and yellowish brown for vibrations perpendicular to the plan of the axes.

The crystals of osmyloxalate, in the dry state are relatively very stable; when heated to about 80° they effloresce and lose their water of crystallisation without the anhydrous salt appearing to undergo the slightest decomposition, as well *in vacuo* and in air, as in hydrogen or carbonic acid. If they are heated more strongly, *in vacuo* to prevent the oxidising action of the air on the probable products of decomposition, it is not until a temperature of about 180° is reached that blackening is observed indicating the commencement of decomposition. This decomposition, which is complete when a dull red heat is reached, furnishes carbonic acid gas only; measurements of its volume show that three molecules of the gas are given off for one molecule of the salt (calculated, 25.7 for the hydrated salt; found, 27.2, 26.9, 27.2, 27.3, 27.1; mean, 27.1).

The black residue from this operation does not contain any metallic osmium, since when heated in a current of hydrogen it forms water-vapour; the collection and weighing of this water show that it is formed molecule for molecule of the salt (calculated, 3.51 for the hydrated salt; found, 3.74); consequently, the osmium in this residue exists in the form of the protoxide. The relative weight of this residue also corresponds closely to that required with regard to one molecule of the salt used, by the formula $\text{OsO} + \text{CO}_2\text{K}_2$ (calculated, 67.3 of the hydrated salt; found, 65.3, 65.5; mean, 65.4). Finally, this residue, either before or after reduction in hydrogen, has a strongly alkaline reaction, is very deliquescent, and effervesces with acids; it contains, therefore, an alkaline carbonate, which appears to be partially dissociated, as shown by the variations in the figures given above from the theoretical

figures. Consequently, the decomposition of the osmyloxalate of potassium by heat can be represented by the following equation:—



Osmyloxalate of potassium is very slightly soluble in cold water, but its solubility is notably increased as the temperature is raised. It is practically insoluble in water charged with chloride of potassium, even when warm.

Solutions thus obtained are very unstable; at about 80° they become cloudy, and give a black deposit of osmic acid, resulting from the reaction,—

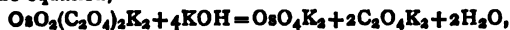


At the same time, the characteristic odour of peroxide of osmium is given off, the presence of which appears to be due to the rapid oxidising power of osmic acid in the presence of air and water, as shown by MM. Moraht and Wischin (*loc. cit.*, p. 156).

The same decomposition takes place in the cold, but much more slowly. It is prevented by the presence of a small quantity of oxalate of potassium, and more so by oxalic acid or a binoxalate, so the decomposition undergone by the salt on contact with pure water would be limited by the presence of the binoxalate formed. In the cold, the amount of oxalic acid necessary to give stability to a concentrated solution of osmyloxalate is very small, 0.1 c.c. of demi-normal oxalic acid solution is sufficient to prevent the decomposition of 100 c.c. of a saturated solution. But this amount must be increased if the solution to be made stable is to be heated; it may also be diminished with the dilution of the solution.

In the presence of a quantity of free oxalic acid sufficient to assure the stability of the solutions obtained, water dissolves about 0.75 per cent of its weight of the osmyloxalate at 15°, and 3 per cent at 50°.

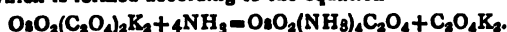
Different to oxalic acid, potash facilitates the decomposition of osmyloxalate of potassium; but the products of the reaction vary according to the manner in which it is effected. If we pour a slightly acid solution of osmyloxalate slowly into an excess of potash-lye, the mixture immediately takes the reddish violet tint of a solution of osmate of potassium, and this salt, formed according to the equation,—



is deposited in the crystalline state on cooling, if sufficiently warm and concentrated solutions have been used.

If, on the other hand, the alkaline lye is added drop by drop to the solution of osmyloxalate, it will be seen that every drop will cause a deep brown colouration at the point where it touches the solution, similar to that which characterises the solutions of peroxide in potash. Even if the movement of the liquid prevents this local formation of colour, the tint of the whole becomes darker with the addition of increasing quantities of the alkali, and gradually tends towards black. At the same time a transitory, attenuated, black precipitate appears, which is formed apparently of osmic acid, and the characteristic odour of peroxide of osmium is also observed. But it is only when a large excess of alkali has been used that the violet tint of solutions of the osmate is observed.

Ammonia, added in equal quantities to a solution of osmyloxalate of potassium, gives a yellow, crystalline precipitate; this is the oxalate of osmydiammonium, discovered in 1881 by Wolcott Gibbs (*loc. cit.*, p. 238) and which is formed according to the equation—



Hydrochloric acid, poured in excess over crystals of osmyloxalate of potassium, attacks them rapidly at about boiling-point, giving off chlorine and forming a yellow liquid, which on cooling deposits regular, deep red octahedra of chlorooximate of potassium (Os calculated, 39.63—found, 40.09; 2KCl, calculated, 30.95—found, 30.37). This transformation may be represented by the equation



All these reactions which connect osmyloxalate of potassium with already known compounds of osmium, form so many indirect proofs of the formula to which the analysis of this salt brings us. Its transformation into osmic acid, or an osmate by water or the alkalis, like its preparation from osmate of potassium and oxalic acid, is effected without any evolution of gas; thus, there are no phenomena of oxidation or reduction in these reactions, and consequently the osmyloxalate is, like the osmate, a hexavalent derivative of osmium. The change brought about by ammonia, and in which the group OsO_2 —characteristic of the osmyl salts is retained, leads to the same conclusion. Finally, its transformation into chlorooximate, a salt derived from quadrivalent osmium, is accompanied by an evolution of chlorine, the estimation of which (calculated 13.8 per cent of the weight of the salt used,—found 14.1) verifies quantitatively the above equation, and thus furnishes a measurement of the difference in valency undergone by osmium in one salt and another.

The constitution of osmyloxalate of potassium being thus well established, it becomes possible to take it as the starting-point for the preparation of other osmyloxalates, and, more generally of other corresponding osmyl salts, like those of the type $\text{OsO}_2\text{X}_2\text{M}_2$.

This research, of which a few results have been already briefly published (L. Wintrebert, "On some Osmyloxalates"), will be the subject of a future paper.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 12.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING SEPTEMBER 30TH, 1902.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, October 10th, 1902.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 200 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Sept. 1st to Sept. 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 200 samples examined by us chemically during the month, all were found to be clear, bright, and well filtered.

Rain has only fallen on seven days at Oxford during the past month, the total fall being 1.11 inches, of which a considerable proportion, viz., 0.85 inch, fell on the 10th and 11th; the average rainfall for September is 2.39 inches. We have thus a deficit of 1.28 inches, which, added to the previous one of 5.35 inches, makes a total deficit for the year of 6.63 inches, or 36.6 per cent on the thirty-five years' average.

Our bacteriological examinations of 533 samples have given the results recorded in the following table; we have also examined 39 other samples, from special standpipes, wells, &c., making a total of 572 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 26 samples) ..	166
New River, filtered (mean of 128 samples) ..	14
Thames, unfiltered (mean of 26 samples) ..	2491
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 293 samples) ..	28
Ditto ditto highest	197
Ditto ditto lowest	0
River Lea, unfiltered (mean of 26 samples) ..	205
River Lea, from the East London Company's clear-water wells (mean of 34 samples) ..	20

Of the 455 samples taken daily from the filter-wells of the Metropolitan Water Companies, and examined bacteriologically by us, sixteen samples, or 3.5 per cent, were sterile. Two samples contained more than 150 microbes, while eighteen samples, or 3.9 per cent, contained more than 100 microbes per c.c. The mean number of microbes in the eighteen excess samples was 128, against a corresponding mean of 142 in twelve samples during August, showing that the purity of the supply has improved during the month of September. In this respect the present autumn resembles those of the last few years in being exceptional in the purity of the River supply, owing to the extraordinarily diminished rainfall.

We are, Sir,
Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

OUR PRESENT KNOWLEDGE OF AROMATIC DIAZO-COMPOUNDS.*

By GILBERT THOMAS MORGAN, D.Sc., F.I.C.

(Continued from p. 191).

C. Diazo-compounds Employed in the production of Azo-colouring Matters.

1. *Aminoazo-compounds*.—The action of a diazonium salt on a primary aromatic amine gives rise either to a diazoamine or an aminoazo-compound, according as to whether the diazo-radicle remains attached to the aminic nitrogen or migrates into the aromatic nucleus. Aniline and its homologues and substitution products yield diazoamines, which are often capable of undergoing re-arrangement into aminoazo-compounds, this change being effected either by allowing the diazoamine to remain in alcoholic solution or by warming it with a mixture of the parent base and its hydrochloride. The latter mode of transformation has been studied quantitatively by Goldschmidt and his pupils (*Ber.*, 1896, xxix., 1369, 1899), with the following results:—

The transformation of diazoaminobenzene into aminoazobenzene in an aniline solution containing aniline hydrochloride is a monomolecular reaction, the velocity of transformation, in moderately dilute solution, being proportional to the temperature and to the amount of catalyst (aniline hydrochloride) but independent of the concentration. Benzenediazoamine-*p*-toluene, when dissolved in aniline containing its hydrochloride, becomes converted into diazoaminobenzene and *p*-toluidine, the resulting diazoamine then undergoing transformation in accordance with the preceding rules.

This conversion takes place more slowly when the diazo-radicle shifts into the ortho-position with respect to the amino-group. The transformation constant of diazoaminobenzene at 45° is 0.081, whereas the value of this coefficient for diazoamine-*p*-toluene is only 0.0095, the solutions employed in both cases being seminormal.

* Report presented to Section B, British Association, Belfast Meeting, 1902.

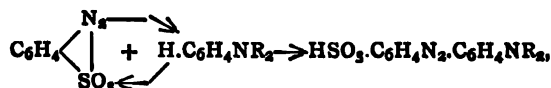
The existence of analogous intermediate diazoamino-compounds and diazohydroxy-derivatives in the commercial azo-colouring matters, has been recently demonstrated by Vaubel (*Zeit. Farben. Textilchem.*, 1902, i., 3).

Although aniline and other benzenoid primary amines give rise to diazoamines when treated with diazonium salts, their dialkyl derivatives containing a free para-position with respect to nitrogen, readily furnish azo-compounds of the methyl-orange type. The formation of these colouring matters is governed by the following laws (Goldschmidt and Merz, *Ber.*, 1897, xxx., 670 and 2075):—

(i.). The velocity of formation of the aminoazo-compound depends only on the nature of the reagents, and not on the concentration.

(ii.). In coupling the hydrochloride of a tertiary base with diazobenzene sulphonic acid, the interaction occurs between the diazo-compound and the base set free by the hydrolytic dissociation of its salt.

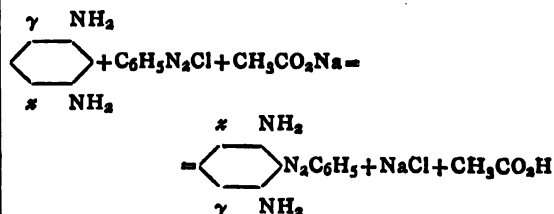
These laws are deduced from the following facts:—The concentration of the hydrochloride of the base or the diazo-compound, has no influence on the velocity of condensation. Excess of hydrochloric acid lessens the velocity. The formation of methyl-orange or the corresponding ethylated colouring matter,—



when carried out in the presence of different acids (e.g., acetic, mono-, di-, and tri-chloroacetic acids and hydrochloric acid), takes place most rapidly with the weakest acid, the velocity decreasing as the affinity constant of the acid increases.

Azo-derivatives of the aromatic *meta*-diamines, although amongst the oldest of the colouring matters, are still manufactured on a large scale for the use of the dyer. Bismarck brown, which was prepared even before the exact nature of the diazo-reaction had been elucidated, is still employed in the arts, and the precise composition of the commercial product has only recently been established by the researches of Täuber (*Ber.*, 1897, xxx., 2111, 2899; 1900, xxxiii., 2116).

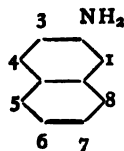
Chrysoidine, first introduced by Witt (*Ber.*, 1877, x., 656), also maintains its position against the newer dye-stuffs. Its homologues and substitution products may be readily and quantitatively prepared from any of the derivatives of *m*-phenylenediamine, providing that these substances contain at least one free para-position with respect to an amino-radicle. If this condition is not fulfilled, the production of an azo-compound, although still possible if the diamine contains a free ortho position with respect to nitrogen, is nevertheless very considerably hindered. The formation of azo-derivatives from symmetrically disubstituted primary *m*-diamines has been demonstrated (Morgan, *Trans.*, 1902, lxxxi., 86), but the reaction—



takes place with considerable difficulty, and the yield of azo-compound is small; moreover, the complete alkylation of the amino-radicles altogether prevents this condensation (*Trans.*, 1902, lxxxi., 650).

In the case of the naphthylamines and their sulphonic acids, the formation of ortho- and para-aminoazo-

compounds takes place with equal readiness; β -naphthylamine,—

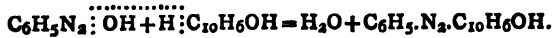


however, only yields azo-derivatives when the positions 1 and 8 are unoccupied. The presence of substituent radicles in either of these positions leads to the formation of diazoamines (Witt, *Ber.*, 1888, xxi., 3483; Morgan, *Trans.*, 1902, lxxxi., 91), and there appears to be no tendency for the diazo-group to migrate into position 3.

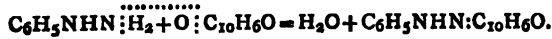
2. *Hydroxyazo-compounds*.—The formation of benzene-azophenol by the action of benzenediazonium chloride on an alkaline solution of phenol is a typical example of the method of preparing this important class of substances. Goldschmidt (*loc. cit.*) found that the essential reagents in this reaction are the diazo-hydroxide and the phenol set free by the hydrolysis of its alkali derivative. Excess of alkali hinders the condensation, the velocity of which diminishes as the concentration of the phenol or diazo-compound increases.

Although at first sight the mode of formation of hydroxyazo-compounds seems to leave little room for doubt as to the constitution of the products, yet this point has, for many years, been the subject of considerable controversy. This discussion arose from the fact that certain of these hydroxyazo-compounds can also be prepared by condensing the aromatic hydrazines with quinones; 4-benzene-azo- α -naphthol, for example, can be obtained either from benzenediazonium chloride and α -naphthol, or from phenylhydrazine and α -naphthoquinone. Similarly, the corresponding 2-benzene-azo- α -naphthol is produced either as a by-product in the former of these reactions, or by the action of phenylhydrazine on β -naphthoquinone, and the alternative methods of formation may be represented in the following manner:—

(i.). Azo-condensation,—

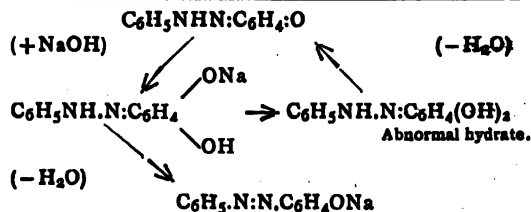


(ii.). Hydrazone condensation,—



Since the two reactions give rise to identically the same substance, it follows that intramolecular re-arrangement must have occurred during one or other or both of these condensations. This problem, which was for some years attacked by purely chemical methods (Liebermann, *Ber.*, 1883, xvi., 2858; Zincke, *Ber.*, 1884, xvii., 3026; 1887, xx., 3171; Meldola, *Trans.*, 1889, lv., 114, 603), has more recently been approached from the physico-chemical side, but even now the question can scarcely be said to be definitely settled. The cryoscopic determinations made by Auwers and his pupils (*Zeit. für Phys. Chem.*, xxi., 355, and *Ber.*, 1900, xxxiii., 1302) indicate that only the ortho-hydroxyazo-compounds are quinone-hydrazones the para-derivatives being phenolic. Chemical evidence in support of the azo-formula for the para-hydroxy-compounds is furnished by Hewitt (*Trans.*, 1900, lxxvii., 99, 712, 810), whilst Noelting's results show that these substances may also react as hydrazones (*Ber.*, 1887, xx., 2997).

The question has again been revived by Hantzsch, who places the hydroxyazo-compounds into the category of pseudo-acids. These are acidic substances which have a configuration differing from that of their salts, tautomeric change taking place during the formation of the metallic derivatives:—



The substance $\text{C}_6\text{H}_5\text{NNH:C}_6\text{H}_4\text{O}$ is a non-electrolyte, and hence its salt, when dissolved, should exhibit hydrolytic dissociation; this, however, is not the case, the metallic derivative behaving as the salt of a negatively substituted phenol. The hydrates of the azo-phenols, isolated by Hewitt (*Ber.*, 1895, xxviii., 799; 1898, xxxi., 2118), contain only half a molecule of water, but the compound $\text{Cl.C}_6\text{H}_4\text{NH.N:C}_6\text{H}_4\text{CH}_3(\text{OH})_2$ has been obtained (*Ber.*, 1899, xxxii., 3089), which corresponds with the abnormal hydrate required by Hantzsch's hypothesis.

The manufacture of hydroxyazo- and aminoazo-compounds has acquired considerable importance, owing to the fact that the products of the action of diazotised benzidine, tolidine, and *o*-dianisidine on the sulphonic acids of the naphthalenoid amines and phenols have the valuable property of dyeing unmordanted cotton—a quality which is also shared by the azo-derivatives of primulin and dehydrothio- β -tolidine, two amines containing sulphur which were discovered by Green (*Trans.*, 1889, lv., 227).

The sodium salt of sulphonated primulin can itself be dyed on cotton, and after being diazotised on the fibre and treated with a solution of a suitable phenol or amine, it gives rise to an insoluble azo-compound which, on account of the method of formation, is called an "ingrain" colouring matter.

These insoluble azo-compounds may also be produced by first impregnating a textile fabric with a phenol, and then treating the material with the solution of a diazonium salt. It was in the course of an investigation on diazo-compounds employed in the production of these "ingrain" azo-compounds that Schraube and Schmidt first isolated the so-called "isodiazo-compounds" (*Ber.*, 1894, xxvii., 520; Badische Anilin und Soda Fabrik, D.R.P. 78874, 80263, 81134, 81202). This important discovery inaugurated a new era of investigation on the diazo-compounds, and led to a revival of the discussion regarding their constitution—a question which had been in abeyance for many years.

II. THE CONSTITUTION OF DIAZO-COMPOUNDS.

A. Metallic Diazo-derivatives.

The alkali isodiazo-oxides, discovered by Schraube and Schmidt, are prepared by adding a solution of diazonium salt to a warm concentrated solution of an alkali hydroxide. The presence of negative radicles in the aromatic nucleus of the diazonium salt causes the transformation to take place more readily; the conversion of *p*-nitrobenzenediazonium chloride is effected, even at -10° , whilst the re-arrangement of the unsubstituted diazonium compound requires a very concentrated solution of alkali hydroxide, and a temperature of $130-140^\circ$. The more acidic diazo-compounds are transformed even by the action of the alkali carbonates (Schraube and Schmidt, and Badische Anilin und Soda Fabrik, *loc. cit.*). It was at first supposed that these isodiazo-oxides were metallic derivatives of the primary aromatic nitrosamines, the transformation taking place in accordance with the following equation:—



This view derived support from the fact that these metallic derivatives showed little or no tendency to yield azo-compounds with alkaline solutions of the phenols

moreover, they yielded nitrosamines of the secondary amines on alkylation.



That the nitrosamine formula is insufficient to account for all the reactions of the isodiazio-oxides was soon shown by von Pechmann, who obtained an oxygen ether, $\text{NO}_2\text{C}_6\text{H}_4\text{N}_2\text{OCH}_3$, from silver iso- β -nitrobenzenediazo-oxide; this methyl derivative has the properties of a diazonium salt, yields azo-compounds with the phenols and a diazoamine with aniline, and it evolves nitrogen on boiling with water, β -nitrophenol being simultaneously produced. It therefore follows that the hydroxide corresponding with these metallic derivatives exhibits the phenomenon of tautomerism. On neutralising a suspension of potassium iso- β -nitrobenzenediazo-oxide with a weak acid, a yellow substance is produced, which at first shows little tendency to form azo-compounds with β -naphthol, or its disulphonic acid, but when left in contact with dilute mineral acid, the product slowly dissolves and the soluble compound obtained has all the properties of the original diazonium salt. This change may be indicated in the following manner:—



This restoration of the capacity for coupling with phenols after acidification was at first considered to be characteristic of isodiazio-derivatives; but the test is not very decisive, since the metallic isodiazio-oxides themselves couple with β -naphthol dissolved in alkali hydroxide, providing that the solution is not too alkaline.

It was next found that potassium benzenediazo-oxide exists in two modifications, the *iso*-form previously mentioned and the normal form, which coupled with the phenols much more readily than its isomeride. This type of isomerism obtains generally amongst the metallic diazo-oxides, but the presence of negative substituents in the aromatic nucleus greatly diminishes the stability of the normal modification. On this account it is usually very difficult to obtain both isomerides in a state of purity.

Isomeric salts have, however, been prepared from diazobenzene sulphonic acid; the normal basic sodium salt, $\text{NaON}_2\text{C}_6\text{H}_4\text{SO}_3\text{Na} \cdot 4\text{H}_2\text{O}$, is obtained by working with cooled solutions, whilst the *iso*-salt, which separates in anhydrous crystals, is produced by heating a solution of its isomeride; they are both strongly alkaline, and are distinguished by their behaviour towards β -naphthol, the former salt readily coupling whilst the latter exhibits this property to a very slight extent. The corresponding potassium compounds have been isolated, and similar pairs of isomeric alkali salts have also been prepared from β -bromodiazobenzene-*o*-sulphonic acid (Hantzsch, *Ber.*, 1895, xxviii., 2002; 1900, xxxiii., 2158; and Gerilowski, *ibid.*, 2317).

Diazo- and Isodiazio-oxides.—When the isomeric diazo-oxides were first investigated there existed a tendency to exaggerate the differences between the isomerides, and they were originally supposed to differ essentially in the following respects:—

- (i.). Capacity for coupling with phenols or ethyl acetate and similarly constituted compounds.
- (ii.). Behaviour towards alkylating, reducing, and oxidising agents.
- (iii.). Interaction with benzoyl chloride and alkali hydroxides.

Production of Azo-derivatives.—The normal diazo-oxides exhibit the greater tendency to combine with the phenols, but in the case of both isomerides the rate of formation of hydroxyazo-compounds is largely dependent on the nature of the aromatic radicle attached to the diazo-group, and the free isomeric diazo-hydroxides couple with phenols even more readily than their potassium derivatives (Bamberger, *Ber.*, 1898, xxviii., 444).

It was at first supposed that the metallic isodiazio-oxides did not condense with ethyl aceto-acetate

(Schraube and Schmidt, *loc. cit.*), differing essentially in this respect from the normal derivatives which had long been known to furnish condensation products with the ester (Japp and Klingemann, *Ber.*, 1887, xx., 3398; *Annalen*, ccxlvii., 190; and Clarisen and Beyer, *Ber.*, 1888, xxi., 1697), but Bülow subsequently showed that the isodiazio-compounds yield mixed aliphatic-aromatic hydrazones or azo-derivatives (*Ber.*, 1898, xxxi., 3122; 1899, xxxii., 197). This correction, although suggesting the structural identity of the normal and isodiazio-oxides, does not assist in deciding between the two formulæ R.N:NOH and R.NH.NO , owing to the uncertainty which still exists with regard to the constitution of the aromatic and mixed condensation products, these substances being regarded by some authorities as azo-derivatives, and by others as hydrazones.

Alkyl Diazo-oxides.—The action of methyl iodide on the metallic isodiazio-oxides indicates that the isodiazio-hydroxide itself is a tautomeric substance, for the potassium salt yields a nitrosamine $\text{R.NCH}_3\text{NO}$, whilst the silver derivative gives rise to an *O*-ether, RN:NO.Me , this substance being also produced from the silver derivative of the normal diazo-hydroxide. This result indicates that the isomeric diazo-hydroxides corresponding with the two silver derivatives both have the same structural formula, R.N:NOH . These *O*-ethers contain the diazo-radicle N:N , for they are all explosive and readily couple with the phenols in alkaline solutions (Bamberger, *Ber.*, 1895, xxviii., 225).

Reduction of the Metallic Diazo-oxides.—The members of both series, when treated with sodium amalgam, yield the corresponding hydrazines, provided that the reaction takes place in excess of alkali hydroxide (Hantzsch, *Ber.*, 1898, xxxi., 340).

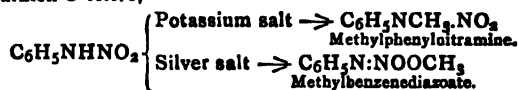
Oxidation of the Metallic Diazo-oxides; the Aromatic Diazoic Acids.—Even before the discovery of the isodiazio-compounds Bamberger had prepared acidic substances by the oxidation of the diazonium salts in alkaline solutions, and on repeating the experiment with the alkali isodiazio-oxides he obtained a larger yield of the oxidation product (*Ber.*, 1894, xxvii., 914). These acidic substances, the aromatic diazoic acids, are also obtained either by dehydrating the nitrates of the primary aromatic amines,—



or by the direct action of nitric anhydride on these bases,—



They readily undergo molecular re-arrangement, yielding nitro-amines, the nitroxyl radicles entering the nucleus either in the ortho- or para-position with respect to the aminic nitrogen (Bamberger, *Ber.*, 1893, xxvi., 471; 1894, xxvii., 359, 584, 2601). These properties indicate that the diazoic acids are nitramines; nevertheless they behave as tautomeric substances, for whereas their alkali salts give rise to secondary nitramines, their silver derivatives furnish *O*-esters,—



The methyl *O*-ester, when boiled with a benzene solution of β -naphthol, yields benzene-azo- β -naphthol, this condensation indicating its relationship to the diazo-compounds.

The Isomeric Diazo-oxides and the Schotten-Beaumann Reaction.—The normal and isodiazio-oxides, when treated with benzoyl chloride, yield nitrosobenzanilide with equal readiness, provided that the reaction is performed in the presence of a large excess of alkali hydroxide. The amount of benzoyl derivative produced is very small when the mixture is only slightly alkaline, the diminution in yield being greatest in the case of the normal isomeride (Hantzsch, *Ber.*, 1897, xxx., 621). This reaction, at first

night, seems to favour the nitrosamine formula for the diazo-oxides,—



but von Pechmann's experiments (*Ber.*, 1892, xxv., 3199) indicate that the product is a tautomeric substance behaving also as if it had the formula—



Thus, when condensed with sodium β -naphthoxide, it yields benzeneazo- β -naphthol, and on treatment with cold potassium hydroxide solution undergoes hydrolysis, giving rise to potassium benzenediazo-oxide, a certain amount of this salt being also formed during the benzylation of the isodiazo-oxide.

Blomstrand, who advocated the diazonium formula—



...

for the normal diazo-oxide (*Journ. Prakt. Chem.*, 1896, liv., 329), supposed that the production of nitrosobenzanilide is due to the transformation of the benzoate—



...

N

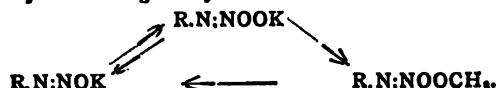
this, however, is not the case, for the ester when prepared from benzenediazonium chloride and silver benzoate does not yield any trace of nitroso-compound under similar experimental conditions (see also Bamberger, *Ber.*, 1897, xxx., 366).

Synthesis of Isodiazo-oxides.—The oxime formula for the metallic isodiazo-oxides also derives support from the fact that the alkali benzene-isodiazo oxide is produced by the action of hydroxylamine on nitrosobenzene in alkaline solutions,—



(Bamberger, *Ber.*, 1895, xxviii., 1218).

Since the isodiazo-oxide may be oxidised to the corresponding benzenediazoate, and reproduced from this substance or its alkyl ester by reduction with sodium amalgam, the cycle of changes may be thus indicated.



B. Stereo-chemical Relationship of the Isomeric Diazo-oxides.

The behaviour of the isomeric alkali diazo-oxides in the reactions described in the preceding section justifies the belief that the two series are structurally identical, and that the comparatively slight differences between the diazo- and isodiazo-derivatives are due to a different spatial arrangement of the radicles associated with the diazo-group, N=N. Accordingly, Hantzsch suggested that this isomerism is comparable with that which obtains among the oximes, the latter kind of isomerism being explained by supposing that the labile and stable modifications contain the associated radicles differently arranged about the complex N=CH in the manner indicated.



The corresponding diazo-oxides would be thus represented:—



(Normal series).

(Iso series).

The assumption involved in both cases is the same—namely, that the third affinity of doubly linked trivalent nitrogen may be exerted in one or other of two definite

directions. If this be granted, then there can be little doubt that the labile normal diazo-oxides possess the *Syn*-configuration, for in this series the coalescence of the contiguous associated radicles readily occurs with the elimination of nitrogen. The stable isodiazo-derivatives should by exclusion have the *Anti*-structure (Hantzsch, *Ber.*, 1894, xxvii., 1702; 1895, xxviii., 676, 1734).

There is one point, however, in which the analogy between oximes and diazo-oxides breaks down; the stereoisomeric oximes give rise to isomeric oxygen-ethers, whereas both the isomeric diazo-oxides yield the same *O*-ether. These *O*-ethers are explosive, and combine with bases and phenols in the presence of alkali hydroxides or carbonates yielding azo-compounds. They are accordingly placed by Bamberger in the *Syn* or normal series (*Ber.*, 1895, xxviii., 225). Hantzsch, on the contrary, points out that in their capacity for forming azo-derivatives they do not differ markedly from the *iso*- or *anti*-diazo-derivatives. The exact relationship of these ethers to the metallic diazo-oxides can scarcely be considered as finally settled.

The *iso*- or *anti*-diazo-oxides undoubtedly exhibit the phenomenon of tautomerism, and under certain conditions, react in accordance with the formula $\text{R}\cdot\text{NH}\cdot\text{NO}$, the formation of *N*-ethers, $\text{RN}(\text{alk})\cdot\text{NO}$ (secondary aromatic nitrosamines), being a case in point.

(To be continued).

PROCEEDINGS OF SOCIETIES.

ROYAL SOCIETY OF NEW SOUTH WALES.

The General Monthly Meeting of the Society was held at the Society's House, No. 5, Elizabeth Street North, on Wednesday evening, September 3, 1902; Prof. WARREN, M.Inst.C.E., Wh.Sc., President, in the Chair.

The following papers were read:—

"*Meteoric Dusts, New South Wales.*" By Professor LIVERSIDGE, M.A., LL.D., F.R.S.

The term meteoric dust is used because it is commonly applied to the materials forming the subject of this paper; it is not intended to state that the dusts are necessarily of cosmic or extra terrestrial origin. The specimens described and exhibited were from Moruya (fell on Dec. 15, 1880); from Uralla (fell on Dec. 14th, 1882); from near Broken Hill (fell 1896); from Menindie (fell June 17th, 1899); and Pambula (fell Oct. 5th, 1899). Dust from the roof-beams and mud from a covered cistern at the University and from the roof of the Observatory, Sydney; all three were collected in 1882. All the dusts are of a reddish colour except those from the University and Observatory, which are grey. The red dusts are mainly siliceous and argillaceous, and look as if they had come from dried-up water-holes; they contain a variety of organic and mineral matters such as might be expected from such a source, and in addition magnetite and metallic iron; the latter contains cobalt and nickel, which seems to indicate that the dusts contain some cosmic or extra-terrestrial materials, part of which may have settled down and become mingled with the undoubted superficial terrestrial deposits and part may have been derived directly from the atmosphere. The University and Observatory dusts also yielded magnetite and metallic iron containing cobalt and nickel, and the University dust yielded particles of gold; the Observatory dust has yet to be tested. The Moruya, Menindie, and Barrier red dusts yielded particles of gold; the others have yet to be examined. Fuller information is given in the paper as to the constituents and chemical composition of the dusts, and analyses of volcanic and other dusts for comparison.

Gold in Meteorites.—Prof. LIVERSIDGE also exhibited under the microscope particles of a malleable yellow metal, which have all the appearance of gold, obtained

from certain Australian and European meteorites (siderolites). The presence of gold in meteorites bears upon the presence of gold in "meteoric" dusts, and it is also of great interest in connection with the presence of gold upon the earth and in sea-water, inasmuch as meteorites and the dust of meteorites are constantly falling upon the earth, to the extent of probably many million tons a year. Further information upon the question of the presence of gold in meteorites will be given shortly in a subsequent paper.

"A Rapid Method of Estimating Lime." By F. B. GUTHRIE, F.I.C., F.C.S., and C. R. BARKER.

The method consists of mixing previously dried and powdered ammonium nitrate with the calcium oxalate precipitate obtained in the usual manner. The oxalate is converted into calcium nitrate, which is very readily and completely converted to oxide by a few minutes' ignition over a Bunsen burner. Prolonged ignition over the blow-pipe is quite unnecessary, and effects no further alteration of the weight of the precipitate. Figures were given showing the accuracy of the method.

NOTICES OF BOOKS.

Inorganic Chemistry, with the Elements of Physical and Theoretical Chemistry. By J. I. D. HINDS. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1902. 8vo., pp. vii.—566, 69 illustrations, cloth.

In the preparation of this volume the author has undertaken to produce "a rather complete text-book on inorganic chemistry for colleges and universities, as well as a handy reference book for all students and teachers of chemistry"; and he "had in mind first an orderly and systematic treatment of the subject without reference to any teaching method."

These ideas have been successfully carried out by Prof. Hinds in this well printed volume, the matter of which is divided into four parts. Part I. contains a general introduction and a statement of its logical divisions; Part II. contains an outline of physical chemistry; Part III. treats more fully of theoretical chemistry; and Part IV. of descriptive chemistry. In classification and treatment the periodic system has been closely followed. A laboratory handbook to accompany this volume is in preparation.

In attempting to give the volume an encyclopædic character the author has enumerated many compounds with very little information concerning each; thus, at page 247, twelve halides and oxyhalides of tungsten are discussed in exactly thirteen lines, and the molybdenum halides at pages 243 and 244 fare little better. At the same time, justice is done to many of the rarer elements, as, for example, the "helioids." Some of the terms employed have an unfamiliar sound, such as vanadoids, scandoids, potassoids, &c.

The reviewer notes that throughout the work the author uses the spelling of chemical terms recommended by the Committee of the American Association for the Advancement of Science—chlorin, bromid, sulfur, &c.

The modern important theories advanced by Arrhenius, Ostwald, van't Hoff, and others are not mentioned in Parts II. or III.; bare reference is made to ionisation at page 97.

The volume can be commended for its very full treatment of the subject in a clear, systematic, and elementary manner.

H. C. B.

Subject-list of Works on the Textile Industries and Wearing Apparel, in the Library of the Patent Office. London: Darling and Son, Ltd., and the Patent Office. 1902. Pp. 127.

This subject-list, which is marked No. 10, of the Patent Office Library Series, is arranged on the same general

plan as the preceding ones already noticed in this column; besides the subjects mentioned in the title, this list also includes works on the culture and chemical technology of textile fibres; the total number of works catalogued is 1138, being 1070 text-books and 68 serials, representing some 1896 volumes in all. The catalogue entries relating to these works number 1513, and are distributed under 185 headings and sub-headings.

Trades' Waste; its Treatment and Utilisation. With Special Reference to the Prevention of Rivers Pollution. A Handbook for Borough Engineers, Surveyors, Architects, and Analysts. By W. NAYLOR, F.C.S., A.M.Inst.C.E. With 21 Plates, 27 Folding Diagrams, and numerous Illustrations in the Text. London: Charles Griffin and Co., Ltd. 1902. Pp. 267.

THE causes and prevention of the pollution of rivers by trades' waste form the principal subject of this volume; the utilisation of trades' waste, except where rivers pollution is concerned, is only touched upon. The fact that the subject is receiving so much attention just now at the hands of engineers, chemists, and bacteriologists must not be attributed to any sudden increase either in the extent or degree of contamination to which rivers have all along been subjected, but rather to a more systematic endeavour to prevent such pollution, so that the rivers may safely serve as a source of domestic water supply to the towns situated along their course.

Sewage is by no means the only source of contamination to which rivers are subjected; woollen mills are great offenders, and as the water arrives at any point where the conditions for erecting a woollen mill are favourable, so does the character of the water commence to deteriorate unless proper preventive measures are taken.

In the *Journal of the Society of Chemical Industry* the various industries are divided into twenty-two classes, of which only five may be said to have no liquid waste of any consequence as to volume, while the remaining seventeen have both liquid and solid wastes, in most cases the liquid predominating. In some cases the provision of settling tanks is sufficient, but it often happens that the deleterious matter is in solution, and must be removed by precipitation and settling, local circumstances deciding all details.

The special trades dealt with by the author in this book are woollen mills, tanning and fellmongering, brewing and distilling, calico-bleaching, printing, and dyeing, paper making, and general chemical waste; these are all taken seriatim, and the various methods of purification proposed and adopted are all fully described and illustrated. There is a good table of contents, and a small index.

The Potash Salts; their Production and Application to Agriculture, Industry, and Horticulture. By Dr. L. A. GROTH, C.E., M.R.I. With a Preface by SAMUEL RIDEAL, D.Sc., Lond. London: The Lombard Press, Ltd. 1902. Pp. 286.

THE deposits of potassium salts are practically limited to German territory, and the recent development in the production, manufacture, and use of these salts has led to the creation of an entirely new and important industry, which in Germany actually competes in success with the coal and iron industries.

In France about 2000 tons per annum of commercial potassium chloride are obtained by the evaporation of sea-water; in India about 20,000 tons of nitrate of potash are obtained annually, and about 1200 tons come from the mines at Kalusz in Galicia, the only potash mine outside Germany. Canada has also exported about 2000 barrels a year, but this quantity has recently fallen off to some extent. These sources, however, are insignificant when compared with the present supply of over 3,000,000 tons a year, for the most part obtained from the great deposits of potash salts occurring naturally at various places in

Germany, and known in general as the Stassfurt potash salts.

There are now twenty potash mines working in Germany, nineteen of which are situated in the northern part of the Hartz district.

It seems fairly certain that these deposits owe their origin to the evaporation of a sea or ocean, but the actual process seems rather obscure, especially as the thickness of the deposits in some places reaches 3000, whereas sea-water containing 3 per cent of solids, even if the depth of the sea had been 30,000 feet, could only have deposited a salt-bed of 450 feet in thickness.

Omitting consideration of the gypsum, anhydrite, and rock-salt, the minerals found in the deposits may be divided, according to their chemical composition and commercial use, into two groups: the potassium and the magnesium salts; in regard to origin they may be classed as primary and secondary salts, the latter including such as have arisen from the solution and re-precipitation of the older deposits. The primary salts are often coloured reddish, the secondary are lighter in colour and much more pure.

It is extremely difficult to distinguish the different minerals by inspection only, and for this purpose, as well as to arrive at the value of the different salts, it is absolutely necessary that chemical analysis go hand in hand with mining.

The crude potash salts, principally carnallite, are partly consumed directly or after pulverisation, while the remainder are transformed into refined and concentrated products as required by chemical industry and agriculture.

The application of potash salts in agriculture is a question of extreme importance. Of the three important classes of artificial fertilisers, viz., nitrates, phosphates, and potash, the two former are generally better understood and more extensively employed than the latter, but the demand for potash is growing steadily, and the consumption for agriculture alone has increased from 71,000 tons in 1890 to 255,000 tons in 1900, calculated in pure potash (K_2O).

A large proportion of the book is devoted to the consideration of agricultural matters, and numbers of experiments with artificial manures are described.

The latter portion of the volume deals with the mining question only; engines, boilers, pumps, rock-drills, compressors, &c., are all described and illustrated, and a good deal of attention is paid to the adoption of electrical machinery both over- and under-ground. Thus the important question of the "potash salts" is dealt with in a thorough manner, both scientifically and commercially, and the whole forms a volume of considerable interest.

tinuous stream under the influence of a minimum electromotive force of about 0.8 volt. The author successively varies the following conditions:—External pressure, concentration of the acid, concentration of the added pyrogallol, and excess of the electromotive force of the pile over the minimum electromotive force necessary for a continuous electrolysis. The external resistance used to obtain the limit at which the electrolytic action ceases to be manifest was varied from very small values up to 1,000,000 ohms. This resistance being measured, and also the electromotive force, the intensity, i , can be calculated from known formulae, and from this the weight of hydrogen, h , evolved per minute can be deduced. Tables are given of the results obtained.

Preparation and Properties of a New Silicide of Vanadium.—H. Moissan and M. Holt.—The authors obtain a new silicide of vanadium, V_2Si , still less fusible in the electric furnace than the silicide VSi_2 . Its composition, density, and colour, besides its action on hydrochloric acid, and its easy decomposition by fusing silicon, are enough to differentiate it sharply from the silicide VSi_2 . The experiments described are a means of establishing the fact that the laws which govern the equilibrium of solutions at ordinary temperatures are equally applicable to the reactions of the electric furnace which are produced between silicon, copper silicide, and vanadium carbide at their boiling-points.

Nitropyromucic Acid and its Ethylic Ether. Di-nitrofurfurane.—R. Marquis.—The use of the nitrating mixture consisting of fuming nitric acid and acetic anhydride having been very successful in the case of furfuran, the author applies it to two other compounds of the same series—ethyl pyromucate and furfural. The ether obtained in the first case consists of yellowish white crystals melting at 101° . These are rapidly decomposed by caustic alkalis, dissolving and giving a red solution which contains an alkaline nitrite. This substance can easily be saponified by heating it in sealed tubes with water to 180° , nitropyromucic acid being thus produced, which melts at 185° .

The Saponification of Nitric Ethers.—Léo Vignon and I. Bay.—The saponification of nitric ethers takes place according to particular rules. These rules are very complex, and they are determined just as well by the easy reduction of nitric acid, first to nitrous acid, then to free nitrogen, and finally to ammonia, as by the oxidation (varying in each case) of the alcohol obtained by saponification.

MISCELLANEOUS.

Monosalicylate of Glycerin.—E. Tauber.—In 1877 Göttig described as monosalicylate of glycerin a substance which he obtained by passing hydrochloric acid gas at 100° through a saturated solution of salicylic acid in glycerin; later on, he described this same substance as being a salicylic dichlorhydrine; this latter view is correct, but the author has observed that though by the action of hydrochloric acid in excess this dichlorhydrine is always obtained, it is sufficient if we wish to prepare the monosalicylate of glycerin, to react with only a small quantity of a mineral acid on a mixture of salicylic acid and glycerin in excess. By heating 100 grms. of salicylic acid, 300 grms. of glycerin, and 12 grms. of sulphuric acid diluted to 60 per cent of acid, for forty hours on the water-bath, the author obtained the ether in question, which, in the pure state, occurs in the form of white microscopic crystals, fusible at 76° . This ether is difficultly soluble in cold water, but easily so in warm water or alcohol; it is very easily saponified by the alkalis and their carbonates; it suffices to leave it in ethereal solution in contact with anhydrous carbonate of sodium or potassium to effect its saponification.—*Berichte*, vol. xxiv., p. 1769.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

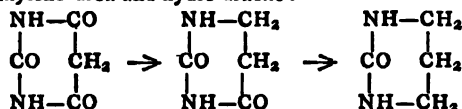
NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxv., No. 13, September 29, 1902.

New Experiments on the Limit of Intensity of the Current from a Pile corresponding to the Manifestation of an Exterior Electrolytic Action apparent in a Voltmeter.—M. Berthelot.—The author determines the limits of intensity of the current from a pile corresponding to the appearance of an exterior electrolytic action. He determines these limits with two different voltmeters: one containing dilute sulphuric acid only, with Wollaston electrodes, a voltmeter in which the production of a reaction continues visible, and there exists a minimum electromotive force of 1.5 to 1.6 volts; and the other containing the same dilute acid added to pyrogallol—a voltmeter in which the hydrogen alone is evolved in a con-

Action of Iodine on Aconitine and Caffein.—C. Klippenberger.—It was thought formerly that the solution of iodine in iodide of potassium formed a periodide of constant composition with the salts of the alkaloids, according to the equation $\text{Alc.CIH} + \text{KI} + \text{I}_2 = \text{Alc.IH.I}_2 + \text{CIK}$. Previous experiments made by the author showed that this equation, as a rule, is inexact, and the present analyses confirm this idea. The practical consequences resulting from this are:—1. Aconitine and caffein cannot be estimated accurately in aqueous solution with an iodine solution, except by titrating this latter by comparison with aqueous solutions of these alkaloids. With caffein, it must be further seen that a notable proportion of the base remains in solution, or at least a large excess of iodine must be added. 2. The use of a solution of iodide of silver in iodide of potassium leads to the formation of a periodide of aconitine having almost exactly the composition $\text{C}_{34}\text{H}_{47}\text{NO}_{11}\text{HI.I}_2$. 3. The volumetric estimation of aconitine by saturation with a titrated acid in the presence of azolithmine, iodeosine, hematoxyline, or cochineal gives more exact results than titration with iodine.—*Zeit. Anal. Ch.*, vol. xxxix., p. 435.

Electrolytic Reduction of Barbituric Acid.—J. Tafel and A. Weinschenk.—The electrolytic reduction of barbituric acid (malonylurea) gives a mixture of trimethylene-urea and hydro-uracil:—



The barbituric acid was prepared by heating an alcoholic solution of urea and soda-malonic ether for a long time at boiling-point; the return was 45 per cent. The reduction was effected in solution in sulphuric acid at 50 per cent, and at about 18–20°. The sulphuric acid was precipitated with baryta, and the filtered liquid evaporated to dryness. The residue was exhausted with cold water, which leaves the hydro-uracil undissolved. This latter crystallises in warm water in flakes, fusible at 274°, subliming at a higher temperature, slightly soluble in the organic solvents, and easily soluble in the fixed alkalis. Hydro-uracil is not attacked either by permanganate of potash in sulphuric solution or by boiling hydrochloric acid. Cupro-ammoniacal solutions oxidise it when warmed. The mercuric derivative obtained by means of HgCl_2 in alkaline solution is crystalline and stable. The argentic derivative (also prepared in the presence of baryta) is in microscopic prisms, decomposed by warm water and by light. Hydro-uracil appears to be somewhat decomposed by boiling baryta. The trimethylene-urea remaining in the mother-liquor can be precipitated in the form of picrate in yellow needles, soluble in 35 parts of warm water; the sulphate occurs in cubical crystals.—*Berichte*, vol. xxxiii., p. 3383.

Analysis of Gutta-percha.—H. Borntrager.—The author has made the following determinations:—1. *Water*.—Desiccation of 2 grms. of material at 100°. 2. *Impurities*.—One grm. of the raw gutta-percha was treated with 50 c.c. of boiling benzene for twelve hours. This was passed through a tared filter and the residue weighed, after washing and drying at 110°. 3. *Pure Gutta-percha*.—The benzenic solution containing the gutta, the fluavil, and the albane, is concentrated to 50 c.c., and precipitated with 100 c.c. of absolute alcohol. After boiling for two hours it is filtered, and the residue dried at 100°, and weighed. 4. *Albane*.—The separate estimation of this substance not being of any technical importance, the sum of the fluavil and the albane is generally determined by difference. However, these two bodies can be separated by concentrating the benzenic solution, containing an excess of alcohol, down to 50 c.c. The albane, insoluble in alcohol, is precipitated on cooling; the decanted solution contains the fluavil. After evaporation and desiccation at 80°, these two bodies are

weighed. According to the author, albane is formed of three almost equal portions, of which the first two distil over at 200° and 250°.

Typical Analysis of a Raw Gutta-percha.

Water		1.5
Impurities		2.5
Pure gutta		77.5
Fluavil		6.0
Albane, a_1		3.8
" a_2		3.7
" a_3		5.0

—*Zeit. Anal. Ch.*, vol. xxxix., p. 502.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Revised Nomenclature.—Could any of your readers inform me if the reform chemical nomenclature mentioned in the *CHEMICAL NEWS* for June 10th, 1892, is generally adopted by the profession, or if not, what parts have been adopted?—C. ELLIOT.

MEETINGS FOR THE WEEK.

FRIDAY, 31st.—Physical, 5. "Existence of a Relationship between the Spectra of some Elements and the Squares of their Atomic Weights," by Dr. W. Marshall Watts. "The Size of Atoms," by H. V. Ridout. Exhibition of "Vacuum Calorimeters," by Prof. H. L. Callendar, F.R.S.

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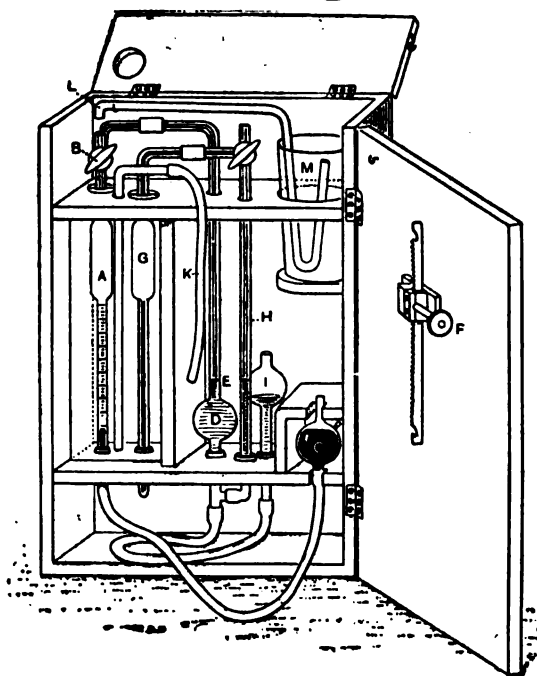
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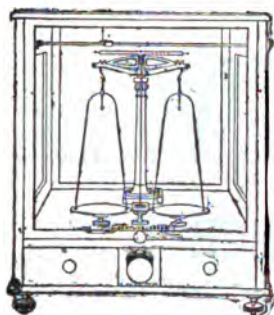
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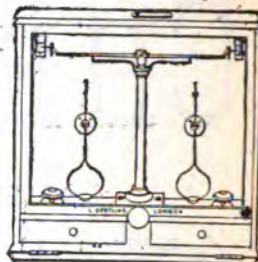


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# THE CHEMICAL NEWS.

VOL. LXXXVI., No. 2240.

## THE MICRO-STRUCTURE OF ZINC AND THE EFFECT OF SMALL AMOUNTS OF IMPURITY ON IT.

By ERNEST A. LEWIS, F.C.S.

THE structure of zinc containing small amounts of different metals, when studied microscopically, gives interesting results, as it shows the effect of small amounts of impurity on a pure metal very well.

In the following experiments chemically pure zinc was melted, along with 0.5 per cent of the various metals, except in the case of cadmium, arsenic, and phosphorus, which were dropped in the molten zinc; the crucibles were then allowed to cool down slowly.

Sections were cut from the centre of each and carefully polished and etched with very dilute nitric acid (1 part of strong nitric acid and 500 parts of water). The remainder of each alloy was re-melted at a low temperature and cast into small cast-iron moulds, and when cold, broken to compare the fracture with the microscopic structure.

*No. 1.*—Pure zinc consists of large primary crystalline grains, and inside the primary crystals, on deeper etching, are seen smaller secondary crystals. The fractured surface shows brilliant large bluish-white crystals.

*No. 2.*—Zinc containing 0.5 per cent of silver consists of small crystals of zinc surrounded by an eutectic alloy of silver and zinc. The fractured surface is finely crystalline.

*No. 3.*—Zinc containing 0.5 per cent of lead consists of both primary and secondary crystals. The primary crystals are similar to those of pure zinc, but the secondary crystals are surrounded by what is probably a solid solution of lead in zinc. The fractured surface is similar to pure zinc.

*No. 4.*—Zinc containing 0.5 per cent of copper is similar in structure to *No. 2*. It consists of crystals of zinc surrounded by an eutectic alloy of copper and zinc; the eutectic alloy is harder than the silver-zinc eutectic, as it shows up on polishing without etching. The fractured surface is finely crystalline.

*No. 5.*—Zinc containing 0.5 per cent of bismuth appears to consist of crystals of zinc surrounded by bismuth. The bismuth does not appear to alloy with zinc. The fractured surface is similar to pure zinc.

*No. 6.*—Zinc containing 0.5 per cent of cadmium consists of small crystals of a cadmium-zinc alloy surrounded by zinc. The fractured surface is very finely crystalline.

*No. 7.*—Zinc containing 0.5 per cent of tin on very slightly etching shows up a network, but on deeper etching it is seen that the tin is part of each crystal; that is, the tin is thrown to the edges of the zinc crystals. The fractured surface is crystalline, but not so coarse as pure zinc.

*No. 8.*—Zinc containing 0.5 per cent of arsenic is similar to pure zinc. The fractured surface is crystalline.

*No. 9.*—Zinc containing 0.5 per cent of antimony consists of primary and secondary crystals. The primary crystals are similar to the crystals of pure zinc, but the secondary crystals are elongated and appear to be surrounded with antimony. The fractured surface shows large crystals similar to pure zinc.

*No. 10.*—Zinc containing 0.5 per cent of iron. The iron separates out in the crystalline form; it does not appear to form a true alloy with zinc. It probably is dissolved by molten zinc, and on cooling it is thrown out

again as crystals of iron. The fractured surface is fibrous.

*No. 11.*—Zinc containing 0.5 per cent of aluminium consists of small crystals of zinc surrounded by an eutectic alloy of aluminium and zinc. The fractured surface is very finely crystalline.

*No. 12.*—Zinc containing 0.5 per cent of manganese consists of primary and secondary crystals. The primary crystals are like those in pure zinc, but the secondary crystals are more elongated than those of pure zinc. The fractured surface is similar to pure zinc.

*No. 13.*—Zinc containing 0.5 per cent of nickel consists of small crystals of a nickel-zinc alloy surrounded by pure zinc. The fractured surface is finely crystalline.

*No. 14.*—Zinc containing 0.5 per cent of phosphorus is very similar to pure zinc, but the crystals are larger. Small quantities of phosphorus are soluble in zinc, without separating out. Phosphorus hardens zinc. The fractured surface is coarsely crystalline.

310, Dudley Road, Birmingham.

## HISTORY OF THE COMMERCIAL MANUFACTURE OF ARTIFICIAL INDIGO.

By H. BRUNK.

THE manufacture of artificial colouring matters owes its existence to purely scientific researches, and its development is intimately connected with the progress realised by science.

The chemistry of artificial colouring materials has always been cultivated in the laboratories of the universities and of the higher schools, and the industry which has received the first gifts of science with enthusiasm follows the works of our great scientific men with sustained interest, in the hope of gathering the fruits of the rich harvest of their work.

But the young industry is no longer content with the gifts which come to it from scientific centres alone; many distinguished men have placed themselves entirely at its service. Such men as Caro, Glaser, Martius, and, later on, Laubenheimer, Duisberg, Bernthsen, and many others, have carried the spirit of scientific research into the domain of practical work. Henceforth, the industry will not be content with *receiving* only, it will also be able to *give*; it will carry out researches and enrich science.

Methods alone do not suffice commercially; processes are needed, and processes capable of being carried out practically. When the results obtained do not satisfy the exigencies of commerce, they must be put on one side, despite their eventual scientific value. Only that work the results of which have a practical value, and which can be effectively patented, is published to the world.

The result is that patents constitute almost the only source from which an idea can be formed of the results obtained from the chemical researches instituted with a practical object.

Unfortunately, patents only describe the results of a research, and do not give, like purely scientific publications, the successive steps which have been followed and those which have been abandoned; all that generally remains unknown. Such an example is indigo, the history of the development of whose manufacture is the task I am to-day confronted with.

The first complete synthesis of a vegetable colouring material was effected in 1868; Graebe and Liebermann pointed out the road which led from anthracene to alizarin, and industry took it up with magnificent success. The industry of the artificial colouring materials obtained a victory which gave it courage to direct its steps towards a still more difficult end—the conquest of the oldest and most important of the vegetable colouring materials, indigo.



The formation of indigo from orthonitroacetophenone was of no commercial use, but after his synthesis of indigo from isatin, completed by the synthesis of this latter substance, Ad. Baeyer, in 1880, effected his beautiful synthesis of indigo from orthonitrophenylpropionic acid. The problem of the manufacture of indigo then took a concrete form.

Orthonitrophenylpropionic acid was a derivative of cinnamic acid. This acid could be prepared from benzoic aldehyde by Perkin's reaction, and benzoic aldehyde again was easily prepared from toluene.

The Badische Anilin und Soda Fabrik and the Farbwerke vorm. Meister, Lucius, and Brüning acquired Baeyer's patents, and commenced, with the help of the inventor, the researches which lasted nearly twenty years, with the object of adapting the process to the requirements of industry.

They first succeeded in preparing cinnamic acid with chloride of benzyl and acetate of soda, instead of using Perkin's method. But from the commencement, orthonitrocinnamic acid was very expensive; the nitration of cinnamic acid by the usual methods only succeeded in transforming a small part of the acid into the orthonitrated derivative, while the greater portion was converted into a parannitrated derivative of no use at all in the manufacture of indigo. Eventually, by using cinnamic ether instead of the free acid, the nitration was effected in such a manner that 70 per cent of the acid was transformed into the orthonitrated derivative.

The bromination of orthonitrocinnamic acid and the transformation of the dibromised acid into orthonitrophenylpropionic acid offered many practical difficulties, but the work carried out with energy and skill resulted, in the spring of 1881, in the manufacture of orthonitrophenylpropionic acid being carried out in a continuous manner. However, the process was not yet able to produce indigo at as low a price as the natural product, and the use of propionic acid was tried in another direction. At this time, indigo printing was the secret of a few houses only, and the idea occurred to effect the transformation of propionic acid into indigo on the fibre itself; this was successfully done when Caro found, in xanthogenate of soda, the appropriate means of reduction, but this success was theoretical rather than practical.

The year 1882 brought the synthesis of indigo by Baeyer and Drewson from orthonitrobenzoic aldehyde and acetone; it was much simpler than that from cinnamic acid, but the rational preparation of the orthonitrobenzoic aldehyde offered apparently insurmountable difficulties; only small quantities of this substance could be obtained by the direct nitration of benzoic aldehyde, together with a large quantity of the meta-nitrated derivative of no value for making indigo.

In 1886 the process of the direct oxidation of methylised benzenes into aldehydes was discovered, without previously transforming them into chlorised derivatives; this roused fresh hopes, and an attempt was made to produce orthonitrobenzoic aldehyde by oxidising orthonitrotoluene, but these experiments were unsatisfactory. Similarly to the process based on the use of propionic acid, the synthesis of indigo from orthonitrobenzoic aldehyde was able to be used partially in 1893, by Kalle and Co., who succeeded in obtaining the intermediate product of condensation, the orthonitrophenylacetic ketone, in the form of its easily soluble bisulphitic derivative. Several years then passed without anything further being published on this subject. It was only in 1896 that it became known that work in this field had been more active than ever.

The Farbwerke de Höchst had developed a commercial method for the transformation of orthonitrotoluene into orthonitrobenzoic aldehyde. This process consisted in treating the mixture of the chlorised products obtained from the chlorisation of orthonitrotoluene by aniline or sulphonic acid, which transformed the chlorised orthonitrobenzyl into orthonitrobenzylamine or the corresponding sulphonised acid. The product is transformed

by oxidation into the benzylidene derivative, which, under the action of acids, splits up into orthonitrobenzoic aldehyde or aniline and sulphonic acid.

At the same time the method of preparing orthonitrobenzoic aldehyde by the direct oxidation of orthonitrotoluene was completed.

Now that the problem of making orthonitrobenzoic aldehyde commercially was solved, the prospects of making indigo from this substance took a more favourable aspect, inasmuch as, through the increasing consumption of paranitrotoluene, there had been for some time an over-production of orthonitrotoluene as a by-product; still there was not enough to make indigo for general use; all efforts were therefore turned towards a synthesis from a raw material which could be obtained in sufficient quantity.

In 1890, Hermann announced that he had obtained indigo by fusing phenylglyccol with caustic potash. This synthesis only required materials that were cheap and plentiful, viz., aniline, acetic acid, chlorine, and alkalis.

However, though the materials were cheap and abundant, the return was too low to make a commercial success. The other glycines, viz., tolylglycine, xylylglycine, &c., behaved in the same manner as phenylglycine. The glycines of the naphthylamines either gave no indigo or not enough to be of any value. Besides, the tint was found not to be equal to that of indigo itself, and did not satisfy the dyers.

However, Hermann had also found that the glyccol of anthranilic acid, phenylglyccolorthocarbonic acid, gave indigo. This method was more satisfactory, and experiments soon showed that it was capable of improvement. But the technical realisation of this synthesis was surrounded by extraordinary difficulties, which sometimes seemed to be insurmountable, and which were only overcome by the efforts of men who joined chemical knowledge with great commercial experience. Happily such men were to be found, and after seven years of hard work they succeeded in solving the problem. This brings me to the development of the manufacture of indigo as now carried out by the Badische Anilin und Soda Fabrik.

Phenylglyccolorthocarbonic acid is obtained by the action of monochloroacetic acid on anthranilic acid. To prepare the latter, it was necessary at first to start from orthonitrotoluene. The orthonitrotoluene could be either oxidised into nitrobenzoic acid, and this reduced; or, on the other hand, the product of the reduction of orthonitrotoluene,—orthotoluidine,—oxidised into anthranilic acid, passing, for example, through the acetyl derivative. This method had the same drawbacks as the formation of indigo from orthonitrobenzoic aldehyde. But it did not stop the undertaking. A method was found by Hoogewerf and van Dorp, by which anthranilic acid could be obtained from phthalic acid.

It was Hofmann who, by his researches on the particular action of bromine in alkaline solution on the acids, gave the idea to the above-mentioned chemists. They succeeded in transforming the imide of phthalic acid into anthranilic acid by means of bromine in alkaline solution.

With phthalic acid as the raw material for the preparation of anthranilic acid, it was naphthalene that became the raw material for the synthesis of indigo. At one stroke the commercial manufacture of indigo was placed on a solid basis. From this moment I had the firm conviction that the method on which we were engaged would bring us to the desired end, which was the substitution of synthetical for natural indigo.

Naphthalene is a raw material for the manufacture of indigo, which can be obtained in practically unlimited quantities. I have calculated that there are 28,000 tons of naphthalene which, for want of other use, are converted annually into lamp-black, or left dissolved in the heavy mineral oils, but which can be isolated at a very cheap rate. This amount is more than sufficient for the manufacture of all the indigo used in the world.

We were also in possession of the best known method for the manufacture of phthalic acid, a process which

consists of oxidising naphthalene with chromic acid, and which was discovered by one of our chemists twenty years ago. But as the cost of this method was too high, it became necessary to find a cheaper means for the oxidation of naphthalene.

M. E. Sapper succeeded in devising an entirely new method for the preparation of phthalic acid; this consisted of heating naphthalene with concentrated sulphuric acid, and a long series of experiments was undertaken with a view of making this process practicable. It was found that the addition of mercury to the sulphuric acid increased the yield of phthalic acid to a satisfactory amount. It is true that chance—the corrosion of a socket containing mercury—was the cause of this discovery, but without this accident the discovery would not have been made. The essential point of the process was the necessity of using very large quantities of concentrated sulphuric acid, and the recovery of this acid, pushed to its furthest point, was one of the conditions of success. If we had effected the recovery of the sulphuric acid in lead chambers the new process would have had no advantages over that based on the use of chromic acid; but here we were able to take advantage of R. Knietsch's method for the manufacture of sulphuric acid. The so-called contact method of making fuming sulphuric acid was brought to such perfection at our works that the formation of sulphuric anhydride by the direct union of the gases from the pyrites ovens with the atmospheric oxygen was more economical than the manufacture in lead chambers.

The manufacture of phthalic acid was thus intimately connected with the new method of making sulphuric acid, as this method makes possible the direct and cheap transformation of the sulphurous acid resulting from the oxidation of the naphthalene into concentrated sulphuric acid. It was thus that we made our process a closed cycle of operations. The atmospheric oxygen can be economically introduced into the naphthalene, and our new method for the manufacture of sulphuric acid became one of the foundations of the manufacture of indigo.

At the same time, we were still endeavouring to cheapen the manufacture of the other substances necessary for the synthesis of indigo.

As the production of the necessary quantity of chloroacetic acid, as well as the oxidation of the phthalimide into anthranilic acid, required large quantities of chlorine, we had to look for a cheap source of this oxidising agent. Already, for the present manufacture of indigo, it is necessary to chlorise nearly 2,000,000 kilos. of glacial acetic acid a year. Neither the Weldon nor the Deacon method suffices for this; the former being too costly and the latter giving too dilute a gas.

At about this time, the electrolytic manufacture of chlorine and of alkaline chlorates was attracting attention, so we looked about for the method most suitable for our wants, and eventually decided on the method brought out by the Elektron manufactory at Griesheim S/M, as being the best. Still the purity of the chlorine produced by this method left something to be desired; however, in our process of the liquefaction of the chlorine we found an excellent means for purifying this gas up to the desired point.

The manufacture of the chloroacetic acid was not without difficulties, but even this, complicated at first, was transformed into a relatively simple operation.

The manufacture of phthalimide, of anthranilic acid, and of phenylglycinorthocarbonic acid itself—the mother-substance of indigo—were not so easy as had been foreseen, and long series of systematic experiments was necessary to determine the most favourable conditions for obtaining the maximum yield of pure acid.

One of the most difficult operations was the fusion on the large scale; that is to say, the transformation of the phenylglycocolorthocarbonic acid by fusion with an alkali into the leuco-derivative giving indigo under the action of the atmospheric oxygen.

The indigo which is deposited in the aqueous solution

of the product of the fusion under the action of the atmospheric air, is crystalline. In cases where extreme fineness of the product is required, as in fermentation vats, the indigo obtained is treated with sulphuric acid, the sulphate obtained is decomposed by water, and thus transformed into an extremely fine powder, which is known in commerce as "indigo S."

I have attempted to give the broad outlines of the history of a new manufacture, and there only remains to point out a few factors which will make the influence of this manufacture felt in other branches of industry, and will even affect the conditions and future of the natural product.

The advantages that synthetic indigo possesses over the natural product have often been discussed; the uniformity, the constant proportion of pure indigo in the artificial product, the total absence of all impurities, the facility with which it can be reduced to an extremely fine state of division, which makes it of great value to the dyer, are all great advantages which it possesses over vegetable indigo. It satisfies the dyer who, for want of exact methods of analysis, had to buy, not according to the intrinsic value, but according to outward signs which were often fallacious.

On account of its great purity, synthetic indigo gives purer shades than natural indigo, and dyeing with artificial indigo has become a most simple and easy matter, while formerly with the natural product it required the experience of long years of practice.

Such is the history of our artificial indigo. It is seen that the new industry is not an unexpected gift from heaven. To bring the solution of the problem to a satisfactory conclusion, it has been necessary for many long years to make use of the intelligence and zeal of a staff of educated men, and that at a time when success seemed by no means certain. The first conditions of the practical synthesis of indigo were furnished by the results of a long scientific research. All the methods of advanced technical skill were at our disposal, and, thanks to the knowledge, the zeal, the energy, and the spirit of duty which animated our chemists, we have been able to accomplish a work which will, we hope, mark a distinct era of progress in civilisation.—*Moniteur Scientifique*, Series 4, vol. xv., No. 711.

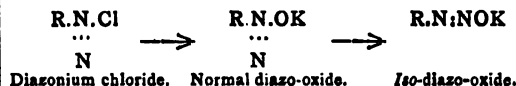
## OUR PRESENT KNOWLEDGE OF AROMATIC DIAZO-COMPOUNDS.\*

By GILBERT THOMAS MORGAN, D.Sc., F.I.C.

(Continued from p. 207).

### B. Stereo-chemical Relationship of the Isomeric Diazo-oxides (continued).

*Structural Formulae for the Isomeric Diazo-oxides.*—The hypothesis that the isomeric diazo-oxides are structurally different has been defended by Bamberger (*Ber.*, 1895, xxviii., 444, 826, 1218) and Blomstrand (*Journ. Prakt. Chem.*, 1896, liii., 169; 1897, liv., 305; 1897, lv., 480), who maintain that the normal diazo-oxide is a diazonium derivative, having the same configuration as the original diazonium salts. There is no longer any difference of opinion with respect to the oxime formula of the isodiazoxides, and accordingly the second hypothesis may be thus graphically illustrated—



One grave objection to the theory is at once detected

\* Report presented to Section B, British Association, Belfast Meeting, 1902.

from this diagram, which indicates that the diazonium hydroxide—



...

N

is capable of acting, not only as an acid, but also as a strong base. The difficulties attending this assumption will be considered at greater length in the sequel.

**Existence of Syn-diazo-Sulphonates, Cyanides, &c.**—Hantzsch, who obtained a very unstable red potassium benzenediazotulphonate isomeric with Fischer's yellow diazo-sulphonate,  $\text{C}_6\text{H}_5\text{N}_2\text{SO}_3\text{K}$ , asserted that these isomerides belonged to the *syn*- and *anti*-diazo-series respectively (*Ber.*, 1894, xxvii., 1726), but Bamberger objected to this classification, and showed that the red salt has the properties of a sulphite,  $\text{C}_6\text{H}_5\text{N}_2\text{O.SO}_3\text{K}$  (*Ber.*, 1894, xxvii., 2930).

The former investigator has, however, succeeded in isolating a series of isomeric diazo-cyanides (*Ber.*, 1895, xxviii., 666). *p*-Chlorobenzenediazonium chloride, when treated with cold potassium cyanide solution, yields a labile salt which readily evolves nitrogen and forms *p*-chlorobenzonitrile on treatment with copper powder, and passes into a stable isomeride. The latter, which is not affected by copper powder and may even be distilled in steam, is undoubtedly the *anti*-diazo-cyanide, whilst the labile derivative belongs to the *syn*-diazo-series. Both isomerides have a yellow colour, and this fact tends to exclude the view that the labile derivative is a diazonium cyanide, for the diazonium radicle gives colourless salts with colourless acids.

The *syn*-diazo cyanides are more stable when the aromatic nucleus attached to the diazo-group contains substituent radicles.

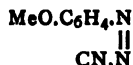
When a diazonium chloride is treated with a suspension of silver cyanide the colourless solution obtained, after filtering off the insoluble yellow *syn*-diazo-cyanide, contains a soluble double cyanide which is considered by Hantzsch and Danziger (*Ber.*, 1897, xxx., 2529) to be a diazonium derivative. The double cyanide derived from *p*-cumenediazonium chloride has been isolated and its properties are best expressed by the formula—



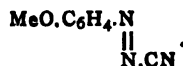
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The *anti*-diazo-cyanide does not yield this double salt.

The formation of this double diazonium cyanide suggests that the sparingly soluble *syn*-diazo-cyanide may exist in solution in a state of equilibrium with the isomeric diazonium salt, and further confirmation of this hypothesis has recently been obtained (*Ber.*, 1900, xxxiii., 2161; 1901, xxxiv., 4166) by a study of the cyanides obtained from *p*-methoxybenzenediazonium bromide and chloride. These salts by double decomposition with potassium cyanide in alcoholic solution yield the *syn*-diazo-cyanide—



an orange-red insoluble substance (melting-point  $51^\circ$ ) which couples with  $\beta$ -naphthol, and slowly changes into the non-coupling *anti*-salt—

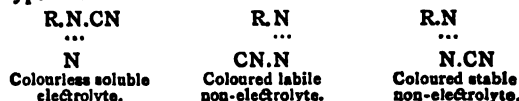


a brownish-red compound melting at  $121^\circ$ .

The existence of a third isomeric cyanide is indicated by evaporating at the ordinary temperature in the presence of excess of hydrogen cyanide an aqueous solution of *p*-methoxybenzenediazonium hydroxide. The colourless, crystalline substance obtained has the composition  $\text{MeOC}_6\text{H}_4\text{N}_2\text{CN.HCN.2H}_2\text{O}$ , and possesses all the properties of a true metallic salt; it is very soluble, and its solution is an electrolyte. Moreover, the double salt con-

denses with  $\beta$ -naphthol and is converted into the yellow *syn*-diazo-cyanide by the action of alkaline solutions.

These results show that diazotised *p*-anisidine gives rise to the three cyanides demanded by the stereochemical hypothesis—



The opposite view only accounts for the existence of two isomeric cyanides, one having the constitution of a normal diazonium salt, and the other that of a diazo-cyanide of the *isodiazo*-series.

The *syn*- and *anti*-diazo-cyanides cannot be regarded as diazonium salts comparable with those of the mineral acids, for on the one hand they are non-electrolytes, dissolving only sparingly in water, and on the other they are distinctly coloured substances, readily soluble in the organic solvents.

Their colour confirms the assumption that they contain the group  $\text{N}=\text{N}$ , in common with azo-compounds. The criterion of colour must, however, be applied with caution, for the *syn*- and *anti*-alkali salts derived from diazotised sulphanilic acid are colourless.

Attempts made to obtain, in a state of purity, *syn*- and *anti*-diazo-derivatives containing other radicles in the place of cyanogen have not proved successful.

The diazonium salts or the alkali *anti*-diazo-oxides, when treated with the alkali phenylmercaptides, yield diazo-thioethers, which do not condense with  $\beta$ -naphthol, and are accordingly taken to belong to the *anti*-series—



...



Only in the case of *p*-chlorobenzenediazothiophenyl ether was an explosive intermediate compound observed which may possibly be a *syn*-derivative. Phenol-*p*-diazo-hydroxide,  $\text{HOC}_6\text{H}_4\text{N}_2\text{OH}$ , and hydrogen sulphide yield a hydrosulphide,  $\text{HOC}_6\text{H}_4\text{N}_2\text{SH.H}_2\text{S}$ , which is possibly an *anti*-diazo-derivative (*Ber.*, 1895, xxviii., 3237).

By treating *p*-nitrobenzenediazonium chloride with hydrogen sulphide, Bamberger (*Ber.*, 1896, xxix., 272) obtained three products:—(i.) A hydrosulphide,  $\text{NO}_2\text{C}_6\text{H}_4\text{N}_2\text{SH.H}_2\text{S}$  or  $\text{NO}_2\text{C}_6\text{H}_4\text{N}(\text{SH}).\text{NH}_2\text{SH}$ , which, like the preceding thio-derivatives, does not couple with phenol; (ii.) a very explosive monosulphide,  $(\text{NO}_2\text{C}_6\text{H}_4\text{N}_2)_2\text{S}$ , condensing with phenols and decomposing in benzene at the ordinary temperature to yield *p*-nitrodiphenyl, hydrogen sulphide, and nitrogen; (iii.) a stable disulphide,  $(\text{NO}_2\text{C}_6\text{H}_4\text{N}_2)_2\text{S}_2$ , which slowly couples with the phenols, and is decomposed by benzene or toluene. The monosulphide is probably a *syn*-diazo-derivative, whilst the disulphide is considered to be an *iso*-(*anti*)-diazo-compound.

**Diazo-anhydrides.**—The isomeric alkali diazo-oxides differ in their behaviour towards cold dilute acetic acid, the *anti*-diazo-oxides giving rise to hydroxides  $\text{R.N}_2\text{OH}$ , which are colourless unless they contain substituent nitro-groups, whilst the *syn*-isomerides furnish extremely explosive yellow diazo-anhydrides (Bamberger, *Ber.*, 1896, xxix., 446). These substances, which may sometimes be obtained by treating a diazonium salt with an alkali *syn*-diazo-oxide, slowly couple with the phenols, yield *O*-diazo ethers with the alcohols, and react explosively with benzene, yielding diphenyl-derivatives. According to Bamberger's earlier papers the anhydrides should have the formula—



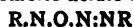
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Hantzsch's stereo-chemical formula (*Ber.*, 1896, xxix., 1067),—



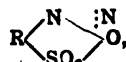
however, explains why the anhydrides readily yield *syn*-diazo-cyanides on treatment with hydrogen cyanide (*Ber.*, 1898, xxxi., 636), but only very slowly dissolve in hydrochloric acid to form diazonium chlorides. Bamberger has since suggested another formula for these substances, which assumes that they are salts of the basic diazonium hydroxide with the isomeric acidic diazo-hydroxide—



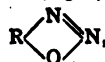
N

This configuration accounts for their formation from a diazonium chloride and an alkali diazo-oxide. The first of these formulae is the least probable, but it is not possible at present to decide between the remaining two.

**Cyclic-Diazo-compounds.**—Diazotised sulphanilic acid and other cyclic diazo-compounds are taken to be either diazonium salts,—



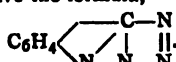
or *syn*-diazo-anhydrides (Hantzsch, *Ber.*, 1895, xxviii., 1734; and *Ber.*, 1896, xxix., 1522),—



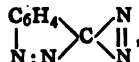
the introduction of negative substituent radicles increasing the tendency to assume the *syn*-configuration. The azimino-derivatives are assumed to be *syn*-compounds,—



The triazoles prepared by Bamberger (*Ber.*, 1899, xxxii., 1773) by the action of nitrous acid on amino-indazoles were assumed to have the formula,—

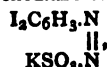


Hantzsch, however, advocates the constitution (*Ber.*, 1902, xxxv., 888)—

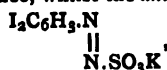


**Diazo-sulphonates.**—These substances, prepared by the action of potassium sulphite on the diazonium chlorides, appear generally to exist in two modifications, but in most cases the *syn*-isomeride is so unstable that it has not been isolated in a pure state. The fact that both modifications are distinctly coloured suggests that they have the constitution  $\text{R.N:N.SO}_3\text{K}$ , analogous to the azo-compounds; the *syn*-isomeride has invariably a more intense colour than the *anti*-salt.

*Syn*-2 : 4-di-iodobenzenediazo-sulphonate,—



is an orange substance, whilst the *anti*-salt,—



is yellow.

The naphthylamines behave exceptionally, yielding only *syn*-diazo-sulphonates; these are not converted into the *anti*-salt on warming, but decompose, yielding the corresponding azo-naphthalenes (Hantzsch and Schmidt, *Ber.*, 1897, xxx., 71).

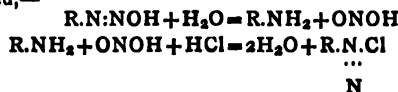
**Diazo-phenylsulphonates** (Hantzsch and Singer, *Ber.*, 1897, xxx., 312).—By the action of benzenesulphonic acid on the diazo-cyanides, diazo-sulphonates  $\text{RN}_2\text{SO}_2\text{C}_6\text{H}_5$  are produced; the action goes immediately with *syn*-diazo-cyanides, but only slowly with their *anti*-salts. The products do not exhibit stereo-isomerism, and probably belong to the *anti*-series; the *syn*-series has not been isolated.

**C. Diazoamines considered as Anti-diazo-compounds.**

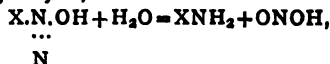
**Isodiazotisation of Aromatic Primary Amines.**—The action of nitrous acid on the salt of an aromatic amine leads to the production of a diazonium salt, the basic nitrogen atom retaining its pentavalency throughout the operation. When the interaction occurs between nitrous acid and the free base, an *iso*-diazo-hydroxide is formed which condenses with a molecular proportion of primary amine, yielding a diazoamine. The direct isodiazotisation of the base may also be effected either by passing nitrous fumes into its solution in dry ether or ethyl acetate (*Ber.*, 1894, xxvii., 1948), or by treating the amine with an alcoholic solution of amyl nitrite and sodium ethoxide (*Ber.*, 1900, xxxiii., 3511); in the former case a mixture of normal and *iso*-diazo-hydroxides results, whilst the latter experiment gives rise to the alkali isodiaz-oxide.

**Migration of the Diazo radicle.**—Griess noticed that a solution of diazobenzenesulphonic acid and *p*-toluidine hydrochloride gave the reactions of a mixture containing *p*-toluenediazonium chloride and sulphanilic acid (*Ber.*, 1882, xv., 2190), and more recently Schraube and Fritsch (*Ber.*, 1896, xxix., 287) showed that a similar rearrangement occurs in the case of a dilute solution of *p*-nitrobenzenediazonium chloride and *p*-toluidine. These investigators state that the direction of the change is constant either in acid or neutral solution; the velocity of migration, however, diminishes rapidly as the amount of acid is increased, and a large excess of this reagent altogether prevents the transformation. These results appear to show that the diazo-radicle passes from the less to the more positive radicle, but Hantzsch and F. M. Perkin found that a migration in the opposite sense occurs with benzenediazonium chloride and *p*-bromaniline (*Ber.*, 1897, xxx., 1412). It may therefore be supposed that generally, when neutral or slightly acid solutions of a diazonium salt and a primary aromatic amine are mixed, there will be a certain amount of rearrangement due to the migration of the diazo-group.

This migration of the diazo-radicle is probably associated with the changes which occur when this group passes from the diazonium to the diazo-configuration. Bamberger (*Ber.*, 1895, xxviii., 826) found that, in the inverse change, a transitory elimination of nitrous acid occurred,—



this reaction being effected by dissolving an alkali *iso*-diazo-oxide in cold mineral acid. If the transitory elimination of nitrous acid is assumed to occur in the change from the diazonium to the diazo-condition, then an explanation of the migration of the diazo-radicle is obtained, which also accounts for the fact that the same mixed diazoamine is produced from the couple  $\text{XN}_2\text{Cl}$  and  $\text{YNH}_2$  as from the combination of  $\text{YN}_2\text{Cl}$  and  $\text{XNH}_2$ . In the former case the diazonium hydroxide, liberated from its salt by the addition of sodium acetate, would undergo hydrolysis,—



and the nascent nitrous acid would at once isodiazotise a portion of each of the primary amines  $\text{XNH}_2$  and  $\text{YNH}_2$ , giving rise to the diazo-hydroxides  $\text{X.N:NOH}$  and  $\text{Y.N:NOH}$ . The latter substances and the unaltered primary bases would then condense, and a mixed diazoamine would be produced, this substance consisting of a solid solution of  $\text{XN}_2\text{NH}_2$  in  $\text{YN}_2\text{NH}_2$ , the proportion of the two constituents depending on the nature of the two aromatic radicles X and Y. In certain cases the product may consist largely of the symmetrical diazoamines  $\text{XN}_2\text{NH}_2$  and  $\text{YN}_2\text{NH}_2$ , but the formation of these compounds is equally well explained by the hypothesis based on the transitory elimination of nitrous acid. The

formation of the mixed diazoamine occurs even when X and Y are radicals derived from different hydrocarbons, and substances of this type containing naphthalene and tetrahydronaphthalene nuclei have been isolated (Morgan, *Trans.*, 1902, lxxxi, 91; and C. Smith, *Trans.*, 1902, lxxxi, 901).

The migration of the diazo-group is prevented by the alkylation of the primary aromatic amine, and the couples  $\text{XN}_2\text{Cl}$ ,  $\text{YNHR}$  and  $\text{YN}_2\text{Cl}$ ,  $\text{XNHR}$  give rise to two isomeric mixed alkyl diazo-amines,  $\text{XN}_2\text{NRY}$  and  $\text{YN}_2\text{NRX}$  (Meldola and Streatfield, *Trans.*, 1887, li, 818; 1889, lv, 610, 1105; 1890, lvii, 785).

The comparative stability of the diazoamines and their production from isodiazoxides point to their having the *anti* diazo configuration, the *syn*-isomerides have not been produced, although Hantzsch and F. M. Perkin (*loc. cit.*) obtained abnormal diazo-amines which form an exception to the rule governing the formation of mixed diazoamines. The compound  $\text{ClC}_6\text{H}_4\text{N}_3\text{HC}_6\text{H}_5$ , for example, exists in two modifications, which give rise to the same products on fission with hydrochloric acid or phenylcarbimide. The suggestion that these two isomerides should be represented by the formulae  $\text{C}_6\text{H}_5\text{N:N.NH.C}_6\text{H}_4\text{Cl}$  and  $\text{C}_6\text{H}_5\text{.NH.N:NC}_6\text{H}_4\text{Cl}$ , is not justified by the result of their fission.

(To be continued).

## THE ESTIMATION OF BROMIC ACID BY THE DIRECT ACTION OF ARSENIOS ACID.\*

By F. A. GOOCH and J. C. BLAKE.

In a former paper from this laboratory (Gooch and Pulman, *Am. Journ. Sci.*, 1901, xii, p. 450), it has been shown that iodic acid may be reduced quantitatively by arsenious acid. In the work of which the present paper is an account the attempt has been made to apply arsenious acid similarly to the quantitative estimation of bromic acid, according to the equation  $3\text{H}_3\text{AsO}_3 + \text{HBrO}_3 = 3\text{H}_3\text{AsO}_4 + \text{HBr}$ .

In the following series of experiments, made to discover the limits within which regularity of action might be expected, definite amounts of arsenious oxide dissolved in acid potassium carbonate were mixed with measured portions of a solution of potassium bromate, sulphuric acid was introduced in the amounts indicated, and, after standing either at the ordinary temperature, on the steam-bath or at the boiling temperature, potassium acid carbonate was added and the arseniate remaining was titrated with iodine to colour, the indication being confirmed by addition of starch. The conditions of acidity, the excess of arsenious oxide, the dilution, the time of action, and the temperature were varied within wide limits. The potassium bromate for this particular series of experiments was thrice re-crystallised from water, dried at  $110^\circ$ , and made up in solution containing 2.8 grms. to the litre. Of this solution 25 or 50 c.m.<sup>3</sup> were measured out for each experiment.

An examination of these results discloses the fact that for conditions varying within a rather wide range the oxidation of the arsenious oxide reaches a fairly definite limit. The bromate effects practically the same proportionate oxidation of arsenious oxide in a volume of 200 c.m.<sup>3</sup> or less, whether the sulphuric acid of half-strength present amounts to 3.5 c.m.<sup>3</sup> or 10 c.m.<sup>3</sup>, and whether the time of digestion is fifteen minutes or thirty minutes at the boiling temperature one and one-half or four hours on the steam-bath, or two days at the ordinary temperature. Setting aside those experiments in which the addition of acid did not exceed the equivalent of alkali carbonate present by more than 1 c.m.<sup>3</sup>, the average

TABLE I.—*Volumes not Exceeding 200 c.m.<sup>3</sup>.*

| KBrO <sub>3</sub> taken.<br>Grm.             | As <sub>2</sub> O <sub>3</sub> taken.<br>Grm. | H <sub>2</sub> SO <sub>4</sub> (1:1) taken.<br>C.m. <sup>3</sup> . | Time of digestion.<br>Mins. | As <sub>2</sub> O <sub>3</sub> un-<br>changed.<br>Grm. | As <sub>2</sub> O <sub>3</sub> oxidised.<br>Grm. | Error, in terms of KBrO <sub>3</sub> .<br>Grm. |
|----------------------------------------------|-----------------------------------------------|--------------------------------------------------------------------|-----------------------------|--------------------------------------------------------|--------------------------------------------------|------------------------------------------------|
| <i>Heated at the Boiling Temperature.</i>    |                                               |                                                                    |                             |                                                        |                                                  |                                                |
| 0.1400                                       | 0.4950                                        | 10                                                                 | 30                          | 0.2476                                                 | 0.2474                                           | 0.0008—                                        |
| 0.1400                                       | 0.4950                                        | 10                                                                 | 30                          | 0.2480                                                 | 0.2470                                           | 0.0010—                                        |
| 0.2400                                       | 0.6188                                        | 10                                                                 | 20                          | 0.3708                                                 | 0.2480                                           | 0.0006—                                        |
| 0.1400                                       | 0.7425                                        | 8                                                                  | 25                          | 0.4941                                                 | 0.2484                                           | 0.0003—                                        |
| 0.1400                                       | 0.7425                                        | 8                                                                  | 25                          | 0.4943                                                 | 0.2482                                           | 0.0004—                                        |
| 0.1400                                       | 0.6188                                        | 7                                                                  | 20                          | 0.3704                                                 | 0.2484                                           | 0.0003—                                        |
| 0.1400                                       | 0.6203                                        | 7                                                                  | 20                          | 0.3720                                                 | 0.2483                                           | 0.0004—                                        |
| 0.1400                                       | 0.6188                                        | 5                                                                  | 15                          | 0.3712                                                 | 0.2476                                           | 0.0008—                                        |
| 0.1400                                       | 0.4950                                        | 3.5                                                                | 15                          | 0.2480                                                 | 0.2470                                           | 0.0010—                                        |
| 0.1400                                       | 0.4950                                        | 3.5                                                                | 15                          | 0.2482                                                 | 0.2468                                           | 0.0011—                                        |
| 0.1400                                       | 0.4950                                        | 2.5                                                                | 15                          | 0.2629                                                 | 0.2321                                           | 0.0097—                                        |
| 0.0700                                       | 0.2475                                        | 5                                                                  | 15                          | 0.1240                                                 | 0.1235                                           | 0.0005—                                        |
| 0.0700                                       | 0.2475                                        | 5                                                                  | 15                          | 0.1239                                                 | 0.1236                                           | 0.0004—                                        |
| 0.0700                                       | 0.2475                                        | 5                                                                  | 15                          | 0.1258                                                 | 0.1217                                           | 0.0041—                                        |
| 0.0700                                       | 0.2475                                        | 5                                                                  | 15                          | 0.1244                                                 | 0.1231                                           | 0.0013—                                        |
| 0.0700                                       | 0.2475                                        | 5                                                                  | 10                          | 0.1239                                                 | 0.1236                                           | 0.0004—                                        |
| 0.0700                                       | 0.2475                                        | 5                                                                  | 10                          | 0.1237                                                 | 0.1238                                           | 0.0003—                                        |
| 0.0700                                       | 0.1584                                        | 5                                                                  | 10                          | 0.0345                                                 | 0.1239                                           | 0.0003—                                        |
| 0.0700                                       | 0.1584                                        | 5                                                                  | 10                          | 0.0357                                                 | 0.1227                                           | 0.0009—                                        |
| 0.0700                                       | 0.1584                                        | 5                                                                  | 10                          | 0.0356                                                 | 0.1228                                           | 0.0009—                                        |
| 0.0700                                       | 0.1584                                        | 5                                                                  | 10                          | 0.0353                                                 | 0.1231                                           | 0.0007—                                        |
| 0.0700                                       | 0.1881                                        | 5                                                                  | 10                          | 0.0649                                                 | 0.1232                                           | 0.0007—                                        |
| 0.0700                                       | 0.1881                                        | 5                                                                  | 10                          | 0.0640                                                 | 0.1241                                           | 0.0002—                                        |
| 0.0700                                       | 0.1881                                        | 5                                                                  | 10                          | 0.0649                                                 | 0.1232                                           | 0.0007—                                        |
| 0.0700                                       | 0.1881                                        | 5                                                                  | 5                           | 0.0652                                                 | 0.1229                                           | 0.0008—                                        |
| 0.0700                                       | 0.1881                                        | 5                                                                  | 5                           | 0.0652                                                 | 0.1219                                           | 0.0008—                                        |
| 0.0700                                       | 0.2475                                        | 2.5                                                                | 10                          | 0.1238                                                 | 0.1237                                           | 0.0004—                                        |
| 0.0700                                       | 0.2475                                        | 2                                                                  | 10                          | 0.1235                                                 | 0.1240                                           | 0.0005—                                        |
| 0.0700                                       | 0.2475                                        | 1.15                                                               | 10                          | 0.1242                                                 | 0.1233                                           | 0.0006—                                        |
| 0.0700                                       | 0.2475                                        | 1.15                                                               | 10                          | 0.1240                                                 | 0.1235                                           | 0.0005—                                        |
| 0.0700                                       | 0.2475                                        | 1.15                                                               | 5                           | 0.1300                                                 | 0.1175                                           | 0.0038—                                        |
| <i>Heated on the Steam-bath —80°.</i>        |                                               |                                                                    |                             |                                                        |                                                  |                                                |
| 0.1400                                       | 0.4950                                        | 10                                                                 | 150                         | 0.2476                                                 | 0.2474                                           | 0.0008—                                        |
| 0.1400                                       | 0.4950                                        | 10                                                                 | 150                         | 0.2476                                                 | 0.2474                                           | 0.0008—                                        |
| 0.1400                                       | 0.4950                                        | 4                                                                  | 240                         | 0.2473                                                 | 0.2477                                           | 0.0007—                                        |
| 0.1400                                       | 0.4950                                        | 4                                                                  | 240                         | 0.2475                                                 | 0.2475                                           | 0.0008—                                        |
| 0.1400                                       | 0.4950                                        | 4                                                                  | 180                         | 0.2472                                                 | 0.2478                                           | 0.0006—                                        |
| 0.1400                                       | 0.4950                                        | 4                                                                  | 180                         | 0.2470                                                 | 0.2480                                           | 0.0005—                                        |
| 0.1400                                       | 0.4950                                        | 3.5                                                                | 90                          | 0.2475                                                 | 0.2475                                           | 0.0008—                                        |
| 0.1400                                       | 0.4950                                        | 3.5                                                                | 90                          | 0.2476                                                 | 0.2474                                           | 0.0008—                                        |
| 0.0700                                       | 0.2475                                        | 1.15                                                               | 30                          | 0.1622                                                 | 0.0853                                           | 0.0217—                                        |
| <i>Digested at the Ordinary Temperature.</i> |                                               |                                                                    |                             |                                                        |                                                  |                                                |
| Hours.                                       |                                               |                                                                    |                             |                                                        |                                                  |                                                |
| 0.1400                                       | 0.4950                                        | 4                                                                  | 46                          | 0.2480                                                 | 0.2470                                           | 0.0005—                                        |
| 0.1400                                       | 0.4950                                        | 3.5                                                                | 41                          | 0.2475                                                 | 0.2475                                           | 0.0008—                                        |
| 0.1400                                       | 0.4950                                        | 3.5                                                                | 41                          | 0.2470                                                 | 0.2480                                           | 0.0005—                                        |
| 0.1400                                       | 0.4950                                        | 3.5                                                                | 17                          | 0.2490                                                 | 0.2460                                           | 0.0016—                                        |
| 0.1400                                       | 0.4950                                        | 3.5                                                                | 17                          | 0.2492                                                 | 0.2458                                           | 0.0017—                                        |

absolute variation from theory of the entire list of forty-two experiments amounts to 0.0007—grm. in terms of bromate, individual variations departing from the average by about the same figure. The obvious meaning of this fact seems to be that the slight error is due to impurity in the potassium bromate employed and not to incomplete oxidising action on the part of the bromate.

In the following series of experiments another preparation of bromate was employed, and its value was fixed by reduction with acidified potassium iodide and titration of the iodine set free according to the method formerly proposed by Kratchmer (*Zeit. Anal. Chem.*, 1885, xxiv, 546). The rate at which the action proceeds according to the equation  $6\text{HI} + \text{HBrO}_3 = \text{HBr} + 3\text{H}_2\text{O} + \text{I}_2$ , has been investigated by Ostwald (*Zeit. Phys. Chem.*, 1888, ii, 127), by Noyes (*Zeit. Phys. Chem.*, 1896, xix, 599), and by Judson and Walker (*Journ. Chem. Soc.*, 1898, lxxiii, 410). Time of action, proportion of iodide to

\* Contribution from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, [4], xiv, No. 82.

TABLE II.

| A.                     |                                     |                          |                   |                                                 |                                |                                  |                       |                                                   |
|------------------------|-------------------------------------|--------------------------|-------------------|-------------------------------------------------|--------------------------------|----------------------------------|-----------------------|---------------------------------------------------|
|                        | KBrO <sub>3</sub><br>taken.<br>Grm. | Iodine<br>taken.<br>Grm. | KI taken.<br>Grm. | H <sub>2</sub> SO <sub>4</sub> (1:1).<br>C.m.s. | Time of<br>standing.<br>Hours. | Approximate<br>volume.<br>C.m.s. | Iodine found.<br>Grm. | Error, in terms<br>of KBrO <sub>3</sub> .<br>Grm. |
| 1.                     | 0.1410                              | 0.6423                   | 1                 | 5                                               | none                           | 150                              | 0.6223                | 0.0044 —                                          |
| 2.                     | 0.1410                              | 0.6423                   | 1                 | 5                                               | none                           | 200                              | 0.5929                | 0.0108 —                                          |
| 3.                     | 0.1400                              | 0.6378                   | 3                 | 5                                               | $\frac{1}{2}$                  | 100                              | 0.6343                | 0.0008 —                                          |
| 4.                     | 0.1400                              | 0.6378                   | 3                 | 5                                               | $\frac{1}{2}$                  | 100                              | 0.6329                | 0.0011 —                                          |
| 5.                     | 0.1400                              | 0.6378                   | 3                 | 5                                               | $\frac{1}{2}$                  | 100                              | 0.6329                | 0.0011 —                                          |
| 6.                     | 0.1408                              | 0.6416                   | 3                 | 5                                               | 22                             | 100                              | 0.6306                | 0.0004 —                                          |
| 7.                     | 0.1408                              | 0.6416                   | 3                 | 5                                               | 22                             | 100                              | 0.6364                | 0.0011 —                                          |
| 8.                     | 0.1408                              | 0.6416                   | 3                 | 5                                               | 22                             | 100                              | 0.6381                | 0.0008 —                                          |
| 9.                     | 0.1400                              | 0.6378                   | 3                 | 2.5                                             | $\frac{1}{2}$                  | 100                              | 0.6340                | 0.0008 —                                          |
| 10.                    | 0.1400                              | 0.6378                   | 3                 | 2.5                                             | $\frac{1}{2}$                  | 100                              | 0.6336                | 0.0009 —                                          |
| 11.                    | 0.1400                              | 0.6378                   | 3                 | 2.5                                             | $\frac{1}{2}$                  | 100                              | 0.6336                | 0.0009 —                                          |
| 12.                    | 0.1400                              | 0.6378                   | 3                 | 1                                               | $\frac{1}{2}$                  | 100                              | 0.6343                | 0.0008 —                                          |
| 13.                    | 0.1400                              | 0.6378                   | 3                 | 0.5                                             | $\frac{1}{2}$                  | 100                              | 0.6331                | 0.0010 —                                          |
| B.                     |                                     |                          |                   |                                                 |                                |                                  |                       |                                                   |
| HCl<br>(sp. gr. 1.18). |                                     |                          |                   |                                                 |                                |                                  |                       |                                                   |
| 14.                    | 0.1400                              | 0.6378                   | 3                 | 8                                               | $\frac{1}{2}$                  | 100                              | 0.6336                | 0.0009 —                                          |
| 15.                    | 0.1400                              | 0.6378                   | 3                 | 8                                               | $\frac{1}{2}$                  | 100                              | 0.6329                | 0.0011 —                                          |
| 16.                    | 0.1400                              | 0.6378                   | 3                 | 8                                               | $\frac{1}{2}$                  | 100                              | 0.6336                | 0.0009 —                                          |
| 17.                    | 0.1400                              | 0.6378                   | 3                 | 4                                               | $\frac{1}{2}$                  | 100                              | 0.6336                | 0.0009 —                                          |
| 18.                    | 0.1400                              | 0.6378                   | 3                 | 4                                               | $\frac{1}{2}$                  | 100                              | 0.6333                | 0.0010 —                                          |
| 19.                    | 0.1400                              | 0.6378                   | 3                 | 4                                               | $\frac{1}{2}$                  | 100                              | 0.6340                | 0.0008 —                                          |
| 20.                    | 0.1400                              | 0.6378                   | 3                 | 4                                               | 1                              | 100                              | 0.6336                | 0.0009 —                                          |
| 21.                    | 0.1400                              | 0.6378                   | 3                 | 4                                               | 1                              | 100                              | 0.6333                | 0.0010 —                                          |
| 22.                    | 0.1400                              | 0.6378                   | 3                 | 4                                               | 1                              | 100                              | 0.6336                | 0.0009 —                                          |

bromate, excess of acid, and dilution are all, within limits, determining factors in the reaction; but in the analytical process it is usually assumed that the reaction goes soon to completion if free acid and a moderate excess of potassium iodide are present. In Table II. are recorded the results of experiments in which measured amounts of standard solutions of potassium bromate (approximately 2.8 grms. to the litre) were treated with potassium iodide and hydrochloric or sulphuric acid for definite times in glass-stoppered bottles, the iodine liberated being determined by titration with sodium thiosulphate standardised against nearly decinormal iodine of value fixed by comparison with decinormal arsenious oxide dissolved in acid potassium carbonate.

In Experiments 1 and 2 the excess of potassium iodide was only about 20 per cent over the amount demanded by theory, the time of standing was only the few minutes required for the manipulation of the process, and the dilution was considerable. It is obvious that under these conditions the reaction is very incomplete. On the other hand, a glance at the table makes it evident that the oxidation in all other experiments, whether in A or B, proceeds to practically the same point, showing similar errors. So it appears that the variation in the amount of acid above the minimum, the kind of acid (whether sulphuric or hydrochloric), and the time above the minimum half-hour, within the limits defined, are without apparent effect upon the reaction in the presence of the amount of potassium iodide used, approximately four times the amount required by theory. The balancing error due to the evolution of iodine by the action of atmospheric oxygen upon the acidified solution of the iodide was found by experiment to vary with the strength of the acid and the time of exposure, from 0.0003 grm. to 0.0003 grm. expressed in terms of the bromate, and these values are not greater than the differences observed between parallel determinations of the same sort. Probably, therefore, all the errors as shown in the table should really be increased a trifle to approximate the truth, notably those of the experiments allowed to stand the longest period, twenty-two hours.

The average apparent error of the process as applied to this particular sample of bromate is 0.0009— grm; and 2.5 c.m.s. of sulphuric acid of half-strength (1:1) or the

equivalent amount of hydrochloric acid, 4 c.m.s. of the acid of sp. gr. 1.18, in the presence of about 3 grms. of potassium iodide, complete the action within a half hour, at a dilution of 100 c.m.s., as far as it will go under any of the conditions tried. The phenomenon noted by Ostwald (*loc. cit.*, p. 131), that small amounts of hydrochloric acid tend to force the reaction more rapidly than equivalent amounts of sulphuric acid, does not appear in these experiments, no doubt because the action was pushed to the limit by the smallest amount of the weakest acid employed.

The error of deficiency shown again appears to be due to impurity in the sample of bromate rather than to incompleteness of the reaction. If the reaction were incomplete it would be natural to look for the cause in the possibility of the inhibiting influence of the iodine set free (Judson and Walker, *loc. cit.*, p. 411), but it was found in three parallel experiments that the introduction of 0.5 grm. of free iodine dissolved in potassium iodide failed to influence the error appreciably. For the present purpose, however, the absolute purity of the bromate is not a matter of moment, if, as seems to be the case, Kratchmer's reaction indicates its value with accuracy, as the basis of experimentation. It seems safe to assume, then, that the average result of Experiments 3 to 22 will give a very fair value for the sample of bromate investigated. That is to say, the experiments show an average deficiency in bromate amounting to 0.0009 grm., or to 0.64 per cent.

(To be continued).

Suboxide of Bismuth.—S. Tanatar.—The oxalate of bismuthyl,  $C_2O_4(BiO)_2$ , is prepared first by digesting  $Bi_2O_3$  with a warm solution containing a known calculated quantity of oxalic acid. The dried salt is calcined as gently as possible in a current of carbonic acid. There remains a black powder of the formula  $Bi_2O_3$ , stable in air, slowly attacked by water, reducing Fehling's solution, and permanganate when warmed. Density at 19° C. = 7.153—7.201. If heated too strongly, a grey powder is obtained, being a mixture of bismuth and  $Bi_2O_3$ . As for the oxalate of bismuth and bismuthyl,  $(C_2O_4)Bi_2O_3$ , it gives a mixture of the suboxide  $BiO$  and the metal.—*Zett. Anorg. Ch.*, vol. xxvii., p. 437.

# A NEW SEPARATION OF THORIUM FROM CERIUM, LANTHANUM, AND DIDYMIUM, AND ITS APPLICATION TO THE ANALYSIS OF MONAZITE.\*

By FLOYD J. METZGER.

THE marked disagreement, among well-recognised chemists, in the determination of thorium in monazite sand, seemed sufficient to warrant a more careful investigation of the methods now existing for its determination, and it was with this object in view that the following work was undertaken.

By thorium, is meant the element as it was generally understood before the more recent researches of Brauner and Baskerville, which point quite conclusively toward the isolation of a new element from what is now generally called thorium.

## Review of Methods.

Among the methods proposed for the separation and purification of thorium, the following may be mentioned as the most important.

Bahr (*Liebig's Ann. Chem.*, 1864, cxxii., 231) was the first to notice that thorium oxalate was soluble in a hot concentrated solution of ammonium oxalate. Since that time numerous investigators, namely, R. Bunsen (*Pogg. Ann.*, 1875, clv., 380), C. Glaser (*Zeit. Anal. Chem.*, 1897, xxxvi., 213), Hintz and Weber (*Zeit. Anal. Chem.*, 1897, xxxvi., 27), and others, have attempted to perfect the separation, and have found that a complete separation of thorium from the other rare earths, in the form of oxalates, cannot be made by one extraction with a hot concentrated solution of ammonium oxalate, but that the operation has to be repeated several times.

The separation of thorium from the other rare earths by using sodium thiosulphate was first proposed by Chydenius (*Pogg. Ann.*, cxix., 46), and later worked by Hermann (*Journ. Prakt. Chem.*, xciii., 106), Drossbach (*Zeit. Angew. Chem.*, 1901, p. 655), Hintz and Weber (*Zeit. Anal. Chem.*, 1897, xxxvi., 676; xxxv., 525), and others.

The separation is based on the fact that thorium is precipitated from a neutral or slightly acid solution by sodium thiosulphate, while cerium and the other rare earths remain in solution.

As was pointed out by Chydenius (*loc. cit.*), and also by Glaser (*Chem. Zeit.*, 1896, xx., 612), the precipitation is not complete in one operation, but by re-precipitating the filtrate with ammonia, converting this into the chloride, and re-precipitating with sodium thiosulphate (repeating as long as a precipitate is obtained with sodium thiosulphate), a complete separation is obtained.

Hintz and Weber (*loc. cit.*) say the separation works best in dilute solutions and in the presence of two or three drops of dilute hydrochloric acid, and give this method preference.

Cleve (*Bull. Soc. Chim.*, 1885, xliii., 53) was the first to show that thorium could be precipitated by hydrogen peroxide from a sulphate solution, and that the precipitate had the composition represented by the formula  $\text{Th}_2\text{O}_5\text{SO}_3$ .

Boisbaudran (*Comptes Rendus*, c., 605) also used hydrogen peroxide as a precipitant about the same time. Wyruboff and Verneuil (*Comptes Rendus*, 1898, cxvii., 340), carried the investigation a little farther, and showed that an analogous precipitate of thorium could be obtained from a solution of the nitrate (*i.e.*,  $\text{Th}_4\text{O}_7\text{N}_2\text{O}_3$ ), and that it gave a complete separation of thorium from the other rare earths.

Kosmann (*Chem. Centrbl.*, 1897, Part 1, 837) separated the greater part of the didymium salt from the crude material, as sulphate. Then he separated thorium from

cerium, lanthanum, and didymium by successive treatment of the acid solution with hydrogen peroxide solution, an acid-ammonium citrate solution, and ammonia. A precipitate of aluminum phosphate and thorium hydroxide was formed, which was afterwards separated by oxalic acid.

Various other methods which are well worth mentioning are:—Urban (*Bull. Soc. Chim.*, 1896, xv., 347) used acetylacetonate of sodium. Chavastelon (*Chem. Centrbl.*, 1900, i., 876) separated thorium by means of sodium sulphite. Boisbaudran (*CHEMICAL NEWS*, 1884, l., 201; also *Comptes Rendus*, 1884, xcix., 525) precipitated thorium, after reducing the cerium, with cuprous oxide. Dennis (*Amer. Chem. Journ.*, 1894, xvi., 79; also *Journ. Amer. Chem. Soc.*, 1896, xviii., 947) used potassium nitride. Delafontaine (*CHEMICAL NEWS*, 1897, lxxv., 230) separated thorium from zirconium by fusion with potassium acid fluoride, and leaching out the double potassium-zirconium fluoride with hot water. Muthmann and Baur (*Ber. d. Chem. Ges.*, 1900, cccxxii., 2028) employed potassium chromate. Kersten (*Pogg. Ann.*, 1839, xlvii., 385) based a method upon the difference in solubility of the oxides in hydrochloric acid. Nilson (*Ber. d. Chem. Ges.*, 1882, p. 2521; 1887, p. 1666) precipitated the hydrated sulphate of thorium.

Undoubtedly the best method which has yet been published for the complete analysis of monazite is that of Glaser (*Journ. Amer. Chem. Soc.*, 1896, xviii., 789).

Benz (*Zeit. Angew. Chem.*, 1902, p. 297) in a recent article, makes a very careful study of the methods now employed for the analysis of monazite (the ammonium oxalate, thiosulphate, and hydrogen peroxide methods), and gives a good comparison of their accuracy.

He finds that one digestion of the mixed oxalates with even a large excess of ammonium oxalate is insufficient to bring into solution all the thorium; on the contrary, he finds that only about one-half the thorium is dissolved, and that after repeated digestions considerable cerium is brought into solution.

With the thiosulphate method he gets very good results after three or four precipitations, and obtains the thorium almost without a trace of cerium. This, however, has the disadvantage of being extremely long.

Hydrogen peroxide, as he shows, precipitates thorium completely from a neutral solution, or from a solution very faintly acid with nitric acid. It also separates thorium from cerium and lanthanum by one re-precipitation.

## EXPERIMENTAL.

### Various Precipitants Tried.

As very few weak organic acids have hitherto been employed for the separation of thorium from its associated rare earths, the following were tried:—

**Maleic Acid.**—Aqueous solutions of maleic acid with thorium, cerium, lanthanum, and didymium give no precipitate hot or cold. Solutions of cerium, lanthanum, and didymium, in 50 per cent alcohol, give nothing hot or cold. A solution of thorium in 50 per cent alcohol gives no precipitate in the cold, but on heating, a partial precipitation results.

**Cinnamic Acid** produces at least a partial precipitation of thorium from 20 per cent alcoholic solutions. The precipitate cannot be filtered (runs through), and for this reason the completeness of the precipitation was not determined. Cerium, lanthanum, and didymium give nothing under the same conditions.

**Picric Acid** and thorium, in alcoholic solutions, give a partial precipitation of thorium. Cerium, lanthanum, and didymium give no precipitate under these conditions.

**Phthalic Acid** in aqueous solution with cerium, lanthanum, and didymium gives nothing cold or hot. Thorium, however, in aqueous solution with phthalic acid gives a partial precipitation on heating. From alcoholic solutions a heavy curdy precipitate results on heating, and is approximately half complete. Cerium, lanthanum,

\* Contribution from the Havemeyer Laboratories, Columbia University. Read at the meeting of the New York Section of the American Chemical Society. From the *Journal of the American Chemical Society*, xiv., No. 10.



and didymium in alcoholic solutions with phthalic acid give no precipitate.

**Fumaric Acid.**—Aqueous solutions, hot or cold, give nothing with cerium, lanthanum, or didymium. Thorium and fumaric acid in aqueous solutions give a precipitate in the cold, which forms slowly, but on applying heat the precipitate forms rapidly (white, flocculent), settles out nicely, and is almost complete. From solution in 95 per cent alcohol, fumaric acid precipitates thorium quantitatively even in the cold. Cerium and fumaric acid in 95 per cent alcohol give no precipitate in the cold, but, on heating, a small fraction of the cerium is thrown down. Lanthanum and didymium in 95 per cent alcohol give nothing cold or hot. From solutions in 40 per cent alcohol, fumaric acid gives a quantitative precipitation of thorium on heating; lanthanum and didymium under the same conditions give nothing. Cerium in 40 per cent alcohol gives, with fumaric acid, nothing cold or hot, unless the cerium solution is quite strong, when a very small fraction is precipitated.

**Ammonium Fumarate** precipitates thorium, cerium, lanthanum, and didymium from aqueous solutions, insoluble in excess, soluble in mineral acids.

Among the amido and amino compounds tried were:—

*Urea* gave nothing.

*Thiourea* gave nothing.

*Acetamide* gave nothing.

*Semicarbasine* gave nothing.

*Succinimide* gives no precipitation of cerium, but gives a partial precipitation of thorium from alcoholic solutions.

*Diethylamine* precipitates thorium, cerium, lanthanum, and didymium.

#### *The Quantitative Precipitation of Thorium by Fumaric Acid.*

From the experiments given on a preceding page it was now evident that thorium could be precipitated quantitatively from alcoholic solutions, leaving cerium, lanthanum, and didymium in solution. It was desirable to know the smallest quantity of alcohol necessary to effect this precipitation, and still leave the cerium, lanthanum, and didymium in solution. This strength was reached when the solution contained 40 per cent of alcohol.

The following results will show the completeness of the precipitation from the various strengths of solution:—

| Alcohol.<br>Per cent. | Volume.<br>C.c. | ThO <sub>2</sub> . |        |
|-----------------------|-----------------|--------------------|--------|
|                       |                 | Taken.             | Found. |
| 15                    | 150             | 0.1568             | 0.1508 |
| 35                    | 150             | 0.1568             | 0.1551 |
| 40                    | 200             | 0.1961             | 0.1962 |
| 40                    | 150             | 0.1568             | 0.1561 |
| 40                    | 120             | 0.1176             | 0.1176 |
| 40                    | 120             | 0.2007             | 0.2003 |
| 40                    | 100             | 0.1004             | 0.1006 |
| 40                    | 120             | 0.1176             | 0.1179 |
| 40                    | 200             | 0.1961             | 0.1962 |

#### *Separation of Thorium from Cerium, Lanthanum, and Didymium.*

The salts used for the experiments were the best nitrates that could be purchased, and the thorium nitrate was purified according to the method suggested by Wyruboff and Verneuil (*Comptes Rendus*, 1898, cxxvi., 4, 340), which is as follows:—

Eighty-three grms. of the nitrate, corresponding approximately to 39 grms. of thoria, were dissolved in 8 litres of water. The solution was divided equally in twelve beakers, and precipitated with hydrogen peroxide (10 c.c. hydrogen dioxide for every 0.5 gm. thorium dioxide). The solutions were then heated to 85° C., and allowed to settle. The precipitates were washed a dozen times by decantation with hot water, and then transferred to a large Büchner funnel, and the washing continued until the filtrate gave no precipitate with ammonia. The precipitate was then transferred to a large porcelain

evaporating dish, and 150 c.c. of concentrated nitric acid added; heat was applied, and the whole went into solution readily. The solution was evaporated to dryness, taken up in water, diluted to 8 litres, and the process repeated. The filtrate from the hydrogen peroxide precipitation was treated with ammonia and hydrogen peroxide, giving a brown precipitate of cerium trioxide. This precipitate of cerium peroxide was examined for lanthanum and didymium, but none could be detected.

A stock solution was made of this purified thorium nitrate and standardised very carefully by precipitation with oxalic acid. This was the thorium used for the experiments. A cold saturated solution of fumaric acid in 40 per cent alcohol (containing approximately 0.1 gm. per 10 c.c.) was used as the precipitant.

(To be continued).

## NOTICES OF BOOKS.

*The New Volumes of the Encyclopædia Britannica.* The Sixth of the New Volumes, being Vol. XXX. of the Complete Work. London: *The Times*. Edinburgh: Adam and Charles Black. 1902. Pp. 845.

THIS volume of the Encyclopædia Britannica comprises those subjects whose titles fall between the alphabetical headings K A B (Kabadian) and M O R (Morvi).

The first article of chemical interest in this volume is one on "Lead," by Mr. H. O. Hofman, Ph.D., in which he draws attention to the important changes in lead-smelting introduced during the last twenty years of the nineteenth century, particularly in America. By the modern method about half the lead is extracted at a very low temperature, leaving rich slags, containing 50 per cent of lead, to be smelted in the blast-furnace, the ultimate result being a very much higher yield than by the other older processes. The reverberatory furnace method, cupelling, the Pattinson and the Parkes process, for smelting and refining lead, are also described.

A long article on "Light," by Mr. C. G. Knott, D.Sc., next claims attention. After a few remarks on the wave theory, we come to photometry and light standards, and here a number of photometers are described. The refraction and dispersion of light are dealt with next, after which we come to polarised light, and the relative motions of matter and æther.

The most important article in this volume, speaking scientifically, is the one contributed by Professor James Dewar, F.R.S., on "Liquid Gases." The article is mainly confined to the discussion of the properties of liquid atmospheric air and the so-called permanent gases—oxygen, nitrogen, and hydrogen—and is a subject Professor Dewar has made peculiarly his own. The article covers twelve pages, and is well illustrated. With regard to the industrial applications of liquid air Professor Dewar does not speak very hopefully. Its use as a source of motive power is, he says, impracticable for any ordinary purpose on the score of inconvenience and expense. Still, in conditions where economy is of no account, liquid air might perhaps, with effectively isolated storage, be utilised as a motive power, e.g., to drive the engines of submarine boats, and at the same time provide a supply of oxygen for the crew.

"Magnetism" is ably treated by Mr. Shelford Bidwell, F.R.S., in an article which contains a short account of the general experimental work in magnetism which has been carried out since the publication of the ninth edition of the Encyclopædia Britannica. Special branches of the subject are "Terrestrial Magnetism," by Mr. Charles Chree, F.R.S.; "Magneto-Optics," by Professor J. J. Thomson, F.R.S.; and "Measuring Instruments," by Professor J. A. Fleming, F.R.S.

In the table of the principal contents of this volume we

observe that there should be an article on "Metallurgy," by Sir W. C. Roberts-Austen, F.R.S.; we are disappointed to find that no such article is given; the more so as metallurgy is a very important English industry. There is a short one, however, entitled "Metallography," by the above-named author, in which he refers to the importance the microscopical examination of metals and alloys has assumed in recent years. A full-page plate is given, containing reproductions of six excellent microphotographs of a gold-copper alloy, three different steels, a gold-lead alloy, and a lead-antimony alloy. The apparatus for taking these photographs is described and illustrated.

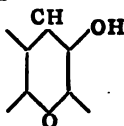
Among the articles of more general interest contained in this volume, we may mention "Military Kites," by Major B. F. S. Baden-Powell; "De Lesseps," by M. de Blowitz; "Lighthouses," by W. T. Douglass; "Machine Guns," by Major H. W. Barlow, R.A.; and "Magic," by J. N. Maskelyne and G. Faur.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

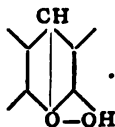
**NOTE.**—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxxv., No. 14, October 6, 1902.

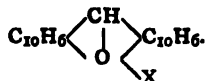
**A Derivative of Hydrogen Peroxide.**—R. Fosse.—In a former paper the author showed that dinaphthapyranol,  $C_{20}H_{16} < \begin{smallmatrix} \text{CHOH} \\ \text{O} \end{smallmatrix} > C_{20}H_{16}$ , in acetic solution possesses a certain oxidising power. The action of dinaphthopyranol on reagents, such as powdered zinc, alcohol, pyrogallol, and the alkaline iodides, shows clearly that this body in acetic solution cannot be considered as an alcohol of pyranol,—



but as a hydrogen peroxide derivative of the hydrate,—



It is known that the first of these peroxides prepared, that of ethyl, was obtained by M. Berthelot, and MM. Baeyer and Villiger have since prepared a great number of peroxides and hydrates of these peroxides. The formula of the author's new peroxide hydrate can be obtained by replacing X by OH in the general formula,—



**Synthesis of certain Tertiary Alcohols.** Diphenylcarbinols.—H. Masson.—The author examines the action of magnesium phenylbromide on various ethers; ethyl formate alone giving a secondary alcohol—benzhydrol,  $(C_6H_5)_2CH.OH$ , melting at  $68^\circ$ . The ethers of the other acids give tertiary alcohols of the form  $(C_6H_5)_3COH$ .—R. These are usually crystalline, and cannot be distilled *in vacuo* without loss of water. Distillation at ordinary pressure leads to the formation of the corresponding ethylenic carbides. The carbides thus produced are not often crystalline; they fix two atoms of

bromine, giving liquid dibromine derivatives. On oxidation they give benzophenone and acids having one carbon atom less than the generating acid. On hydrogenation by alcohol and sodium the corresponding saturated carbides are formed.

**Anhydrous Cupro-ammonium Sulphates.**—M. Bouzat.—The author examines the cupro-ammonium sulphates in order to compare their heats of formation with those of the chlorides. It is already a known fact that the different cupric salts dissolve, evolving the same quantity of heat, when combining with ammonia, and that this relation shows the existence of complex radicals transporting themselves without alteration from one ammonium salt to another. The author now applies the same theory to solids. For this purpose, he prepares various cupro-ammonium sulphates and measures their heats of formation.

**Researches and Estimation on the Extract of Chestnut Tree mixed with Extract from Oak Tree.**—Ferdinand Jean.—When a solution of the extract of chestnut wood is agitated with a solution of iodic acid in the cold a certain quantity of iodine is liberated, whilst with an extract of oak wood this phenomenon does not take place. The reaction is equally negative with solutions of mangrove, mimosa, sumac, mastic tree, fustic, and thorn; logwood is an exception, and liberates a small quantity of iodine. The extracts of oak wood prepared for tanning being frequently falsified with an extract of chestnut wood, it is interesting to find a means of detecting this fraud, which hitherto has not been punishable, owing to the fact of there being no chemical means of recognising it.

**Pectic Fermentation.**—M. Goyand.—When certain vegetable juices are added to a concentrated aqueous and neutral solution of pectine, the mixture becomes a gelatinous mass. This phenomenon is produced by the diastase pectase. It is known that pectase only acts in presence of calcium salts and in a slightly acid solution. The author's present research is to prove that, in the absence of calcium salts, pectase has an action on pectine, and he finds that it transforms pectine into pectic acid, even in the absence of calcium salts. The phenomenon is visible, on account of the formation of insoluble calcium pectate. If, however, the lime is replaced by potash, in the form of potassium oxalate, a potassium pectate soluble in water is produced. These experiments were undertaken with the purpose of finding under what conditions the lime is precipitated by the oxalate of sodium and ammonium. The results are similar in all cases. Another means of verifying this fact is that a neutral molecule of pectine gives one or more molecules of pectic acid, and the acidity of the mixture increases. To verify this hypothesis, it is necessary to use small proportions of pectase, in order that the transformations may not be too rapid. A table of the results obtained is given, and from them the definite conclusion is drawn that pectase is formed from pectic acid by means of pectine, and that the phenomenon is not influenced qualitatively by the presence or absence of calcium salts.

*Bulletin de la Société Chimique de Paris*  
Series 3, Vol. xxvii., No. 7.

**A New Gaseous Body: Hexafluoride of Sulphur,  $SF_6$ .**—H. Moissan and P. Lebeau.—A preliminary experiment of placing a fragment of sulphur in contact with fluorine gas showed that at least two new compounds were formed:—(1) A gaseous body, on which water is without action and which is absorbed by a solution of potash; (2) a gaseous body non-absorbable by water and alkaline solutions, and decomposed by sodium vapour. By using an excess of fluorine the unabsorbable gas is the one formed in the greatest quantity; it may even reach 80 or 90 per cent. It is easily obtained in a state of purity by treating the mixture of the two fluorides with a solution

of potash. As this new body was produced in an excess of perfluorine, it was reasonable to suppose that it would be a perfluorised compound of sulphur; this was proved to be the case. Hexafluoride of sulphur,  $\text{SF}_6$ , is a colourless, odourless, tasteless, incombustible gas, and does not support combustion. It solidifies at about  $-55^\circ$ , forming a white crystalline mass. The gas is very slightly soluble in water, and slightly soluble in boiled anhydrous alcohol. Although so rich in fluorine, it is curious to observe that it is, as a rule, inert, and its properties are comparable with those of nitrogen. Oxygen only reacts on this gas at the temperature of a strong induction spark, sulphur is without action at its fusing-point, though its vapour attacks the gas. Vapour of selenium decomposes fluoride of sulphur, but the reaction is not very definite. At the temperature of softening glass sulphuretted hydrogen reacts easily, depositing sulphur and producing white fumes, which condense, forming colourless drops. Ammonia-gas, either cold or at a red heat, has no action on fluoride of sulphur.

**Density and Analysis of Hexafluoride of Sulphur.**—H. Moissan and P. Lebeau.—The density of this perfluoride of sulphur was determined by means of the apparatus devised by MM. Moissan and Gautier (*Ann. Chim. Phys.*, Series 7, vol. v., p. 56), and the following figures were obtained:—4.95, 4.99, 5.00, 5.11; the theoretical figure for  $\text{SF}_6$  being 5.06. The fluorine was estimated gravimetrically by decomposing the gas with metallic sodium vapour and estimating the fluorine in the fluoride of sodium formed. The figures obtained were—78.40, 78.62, 79.10; the theoretical figure for  $\text{SF}_6$  being 78.08. The estimation of the sulphur gave 22.00 and 22.25, against the theoretical figure 21.91 for  $\text{SF}_6$ .

**Preparation, Properties, and Analysis of Fluoride of Thionyl.**—H. Moissan and P. Lebeau.—Fluoride of thionyl is prepared by passing fluorine gas over chloride of thionyl, the temperature being kept down to about  $0^\circ$ . The flask in which the reaction takes place is connected with another one kept at  $-80^\circ$ , in which the fluoride of thionyl is liquefied. The gas thus obtained is not pure, but contains a little chlorine, which can be removed by agitation with mercury; it also contains oxyfluoride of sulphur, which can be eliminated by a series of liquefactions and fractional distillations. Fluoride of thionyl is a colourless gas, fuming slightly in moist air, with a suffocating odour as disagreeable as that of oxychloride of carbon. Its boiling-point is about  $-32^\circ$ . Its density in three determinations gave 3.04, 2.90, 2.88; theory gives 2.97. It is soluble in chloride of arsenic, essence of turpentine, and benzene. The action of heat on fluoride of thionyl in the presence of glass enables us to determine the composition of this gas volumetrically. Four volumes of  $\text{SOF}_2$  ought to give four volumes of  $\text{SO}_2$  and two volumes of  $\text{SiF}_4$ .

|                  | Volume of gas. | $\text{SiF}_4$ . | $\text{SO}_2$ . |
|------------------|----------------|------------------|-----------------|
| Found .. ..      | 6.8            | 3.1              | 6.1             |
| Calculated .. .. | 6.8            | 3.3              | 6.3             |

A known volume of fluoride of thionyl is treated with water. When the absorption is complete, the liquid is treated with a titrated solution of iodine, and the sulphur estimated. In the liquid, the fluorine is precipitated as fluoride of calcium; at the same time a little sulphate of calcium is deposited. The mixture is dried and weighed, then converted entirely into sulphate, by which means the fluorine can be determined. The following figures were obtained:—

|                |       |       | Theory. |
|----------------|-------|-------|---------|
| Fluorine .. .. | 44.10 | 44.40 | 44.1    |
| Sulphur .. ..  | 36.30 | 36.85 | 37.1    |

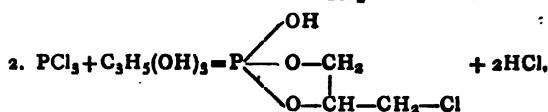
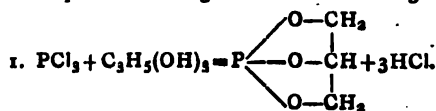
**A New Oxyfluoride of Sulphur: Fluoride of Sulphuryl,  $\text{SO}_2\text{F}_2$ .**—H. Moissan and P. Lebeau.—Already noticed.

**Electrolytic Preparation of Antimonide of Lithium, and Some Alloys of this Metal.**—P. Lebeau.—Already noticed.

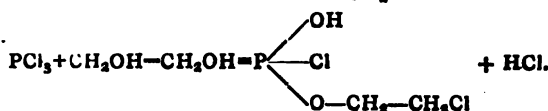
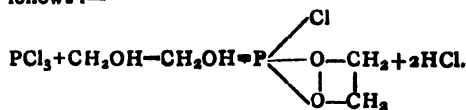
**A new Preparation, and the Properties of Antimonide of Lithium.**—P. Lebeau.—Already noticed.

**Etherification of Phosphorous Acid by Glycerin and Glycol.**—P. Carré.—Already noticed.

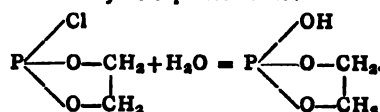
**Action of Trichloride of Phosphorus on Glycerin and Glycol.**—P. Carré.—The author is of the opinion that the reaction of trichloride of phosphorus on glycerin takes place according to the two following equations:—



An analogous research has led him to the conclusion that the action of trichloride of phosphorus on glycol is as follows:—



Only the first of these ethers has been examined; its action on water may be expressed thus:—



**Some Derivatives of *p*-Sulphochloride of Toluene and of *o*-Nitro-*p*-sulphochloride of Toluene.**—F. Reverdin and P. Crépieux.—Already noticed.

**Action of Oxidising Agents on Pentachlorophenol.**—E. Barral.—By oxidation with fuming nitric acid, hexachlorophenol-*a* is transformed integrally into tetrachlorised quinone or chemically pure chloranil; but the author found that, not only did none of the principal oxidising agents transform pentachlorophenol into quinone, but many of them did not give chloranil, while others destroyed the pentachlorophenol to a great extent, forming a minimal fraction of chloranil.

**Transformation of Pentachlorophenol into Tetrachlorised Quinone.**—E. Barral.—By reacting with oxidising agents on pentachlorophenol the author observed the following facts:—1. Whenever chloranil is formed, most of the pentachlorophenol is destroyed. 2. When chloranil is formed, its formation is preceded by that of hexachlorophenol-*a*, on which the oxidising agent reacts, transforming the ketone into diketone. 3. On passing chlorine through hydrochloric acid holding pentachlorophenol in suspension in the cold, he obtained another intermediate body—heptachlorophenol-*a*,  $\text{C}_6\text{Cl}_7\text{OH}$ , fusible at  $98^\circ$ , decomposing at  $130^\circ$  into hydrochloric acid and hexachlorophenol,  $\text{C}_6\text{Cl}_6\text{OH} = \text{C}_6\text{Cl}_5\text{OH} + \text{HCl}$ .

**Examination of the Essence of Sweet Orange Blossoms.**—E. Theulier.—Already noticed.

**Catalytic Properties of the Hydrogenases. Identification of M. Loew's "Catalase" and of M. de Rey-Pailhade's "Philothon."**—The result of a very long paper is to show that these two diastases are identical they have a characteristic reaction, viz., they reduce ferricyanide of potassium to which ferric chloride has been added.

## MISCELLANEOUS.

**F. E. Becker and Co. (W. and J. George, Ltd., Successors).**—We have just received the new Illustrated Catalogue and Price List of Balances, Scales, and Weights issued by this well-known firm. Messrs. George, Ltd., are not only importers of these goods, but are also manufacturers, and undertake repairs and adjustment of the same. This catalogue is very well got up, and it is claimed by the firm to be the largest and best illustrated catalogue in the world devoted entirely to balances and weights. The firm is one of world-wide reputation, and we hope their success will continue. We would call attention to the special balance, illustrated on page 60, manufactured according to Mr. Kuhlmann's design; this balance is constructed with mechanism for rapidly placing and removing the weights; it will carry 200 grms. in each pan, and is sensitive to  $\frac{1}{100}$ th of a m.grm. when fully loaded. On the right-hand side of the case protrude a number of transporters, one for each weight, each being plainly marked with the weight it carries; by this means any number of weights can be placed on, or removed from, the pan very rapidly, while the case remains closed the whole time. By means of an automatic arrangement, on placing a weight on the pan the figure corresponding to its value appears at the side, and disappears when the weight is removed, so that the final reading can be made accurately and almost at a glance.

**Synthetic Formation of Ammonia.**—E. Baur.—The electrolysis of an aqueous solution of nitrate of ammonium saturated with ammonia gas (Divers's solution) gives, at the two poles, hydrogen and nitrogen respectively in the proportion of 3 to 1. Inversely, if we set up a Grove's cell with these two gases over the Divers solution, we observe a tendency towards their re-composition, shown by an electromotive force of 0.6 volt. The phenomena are very similar if we replace the  $\text{NO}_3\text{NH}_4$  by  $\text{KCl}$ . The author has also examined the variation of the electromotive force with the temperature. No trace of ammonia was found by heating the mixture  $\text{N}+3\text{H}$  with platinum black, or with nitrides of chromium or molybdenum, or by leaving the same mixture, in the cold, in contact with platinum foil and a little hydrochloric acid.—*Berichte*, vol. xxiv., p. 2385.

**Quadrantoxide of Cadmium.**—S. Tanatar.—Oxalate of cadmium is calcined at as low a temperature as possible in a current of dry carbonic acid, taking care to stir from time to time; the operation is stopped when no more gas comes off, and the material allowed to cool in the carbonic acid. A beautiful green powder remains, stable in dry air, oxidised slowly by cold water, and decomposed by acids or by ammonia into metallic cadmium and  $\text{CdO}$ . Its formula is  $\text{Cd}_4\text{O}$ , and its density at  $10^\circ$  is 8.177–8.207. If heated too strongly during its preparation, a yellowish brown powder is obtained, which is a mixture of the metal and the protoxide.—*Zeit. Anorg. Ch.*, vol. xxvii., p. 432.

## NOTES AND QUERIES.

\* Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

**Sodium Bismuthate.**—I should like to know how "sodium bismuthate" (mentioned in *Chem. News*, lxxix., p. 209), for oxidation of manganese, is prepared, as I cannot find it in any of the ordinary text-books.—S. C.

**Potassium Persulphate.**—An attempt was made to prepare potassium persulphate by electrolysis of a saturated solution of  $\text{KHSO}_4$  (Kahlbaum's pure) with a thermopile, which gave a current of 4 amperes; the experiment was allowed to continue for 124 hours (overnight), but at the end of that time absolutely no persulphate was formed. The  $\text{KHSO}_4$  solution was placed in a platinum dish, which was surrounded by cold water; in the platinum dish was placed a small porous pot containing dilute  $\text{H}_2\text{SO}_4$ ; the dish was connected

with the anode by a piece of clean platinum, and dipping in the sulphuric acid was a similar piece of foil connected with the cathode; there was a good contact between the dish and foil. Would be very much obliged if some kind reader would give probable cause of failure, or a simpler and better method (if any) of preparing small quantities (20 to 40 grms.) of potassium persulphate, or even ammonium persulphate—the potassium salt preferred.—S. C.

## MEETINGS FOR THE WEEK.

**MONDAY, Nov. 3rd.**—Royal Institution, 5. General Monthly Meeting. Society of Chemical Industry, 8. "Preliminary Investigation of the Chemical Changes produced by various Reagents on Gutta-percha," by Sir William Ramsay. "The Reduction of Ammoniacal Silver Solutions by Organic Substances," by Dr. G. T. Morgan. "A Simple Qualitative Test for Bromides and Iodides," by Dr. F. Mollwo Perkin. "The Influence of Impurities on the Specific Gravity of Sulphuric Acid," by Arthur Marshall.

**WEDNESDAY, 5th.**—Society of Public Analysts, 8. "Reactions of the Alkaloids of Ipecacuanha," and "The Analysis of Preparations containing Opium," by Alfred H. Allen, F.I.C. "Estimation of Salicylic Acid," by Sidney Harvey, F.I.C. "Volatility of Aqueous Solutions of Acetic Acid," by Wm. Chataway, F.I.C.

**THURSDAY, 6th.**—Chemical, 8. "Di-indigotine," by J. Moir. "Localisation of Phosphates in the Sugar-cane," by C. H. G. Sprankling. "Specific Heats of Gases," by H. Crompton. "Non-existence of the Gaseous Sulphide of Carbon described by Deniger," by E. J. Russell and N. Smith. "Action of Nitric Acid on Bromophenolic Compounds," by W. Robertson. "Hydroxyoxamides, Part II," by R. H. Pickard, C. Allen, W. A. Bowdler, and W. Carter. "3:5 Dichlor-o-xylene and 3:5 Dichlor-o-phthalic Acid," by A. W. Crossley and H. R. Le Sueur. "Isometric Anhydrous Sulphates of the Form  $\text{M}(\text{SO}_4)_2 \cdot \text{R}_2\text{SO}_4$ ," by F. R. Mallet. "Catalytic Racemisation of Amygdalaine," by J. W. Walker. "Combination of Carbon Monoxide with Chlorine under the Influence of Light," by G. Dyson and A. Harden. "Constituents of Commercial Chrysarobin," by H. A. D. Jowett and C. E. Potter.

## APPOINTMENT OF EDITOR.

The Council of the CHEMICAL SOCIETY invite applications for the office of EDITOR. The duties will be to Edit the "Journal" and "Proceedings" of the Society and to supervise the preparation, by the Sub-Editor and Abstractors, of the Abstracts of foreign Papers published in the "Journal."

The appointment will be made from 1st January, 1903. Applications, accompanied by full particulars, must be received before WEDNESDAY, NOVEMBER 12, 1902, addressed to the Hon. Secretaries, Chemical Society, Burlington House, Piccadilly, London, W., from whom further particulars may be obtained.

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Particulars of the proposed amendment were set forth in the Illustrated Official Journal (Patents), issued on the 22nd October, 1902. Any person, or persons, may give Notice of Opposition to the Amendment (on Form G), at the Patent Office, 25, Southampton Buildings, London, W.C., within one calendar month from the date of the said Journal.

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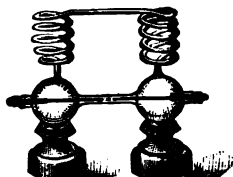
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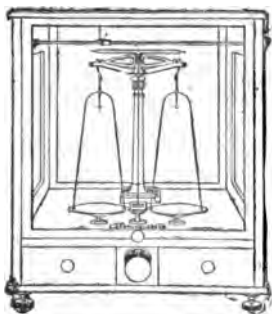
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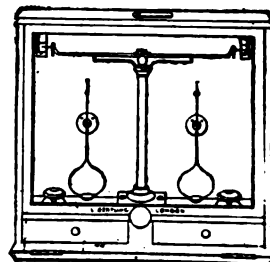


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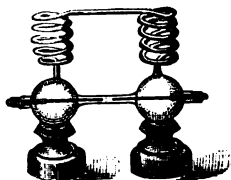
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# THE CHEMICAL NEWS.

VOL. LXXXVI., No. 2241.

## LABORATORY NOTES.

By HORACE JERVIS.

**Carbon by Combustion.**—The estimation of carbon in metals by combustion is perhaps the most elaborate assay the iron-works analyst ventures upon. It is much more elaborate as a matter of tradition than it need be as a matter of fact, and this to some extent accounts for its entire absence from some works, and its meagre practice in some colleges professedly fitted with the most modern apparatus for the rapid and accurate analysis of iron and steel.

The only alternative to carbon by combustion is the Eggertz colour test, whose advantageous use is strictly limited to comparative cases, and whose indications are considerably vitiated or entirely upset by varying amounts of elements—other than carbon—which occur commonly in steels. It is a matter of some moment, therefore, to reduce the combustion process to its simplest forms.

**Changing Boats.**—Boats containing the carbonaceous residue are sometimes changed while the tube is at full redness, so as to economise the time needed for the actual combustion. There is a slight back pressure immediately the stopper is replaced, but it occasions no loss if everything is done smartly. If desired, the aspirator can be attached and allowed to act during the changing, as the small amount of unpurified air thus drawn into the tube has no significant effect on the result; e.g., two and a-half litres of such air increased the weight of a potash bulb 3½ m.grms. only. The boats should be dry and warm when taken from the bath, so as to lessen the danger of cracking the tube. To lower the temperature of the entire furnace with the same object is a bad practice, as it allows CO to pass the barely red CuO without being oxidised.

**Copper Oxide.**—Copper oxide, kept at a red heat, is essential whenever the carbonaceous residue is burned slowly at increasing temperatures; but if the boat is pushed at once into a tube heated to bright redness, no appreciable amount of carbon monoxide is formed, and the copper oxide may be omitted. It is not desired to suggest that the author prefers to work with an entirely empty combustion tube, but merely to state that the results of a series of estimations made in that way varied in no case more than 0.02 per cent from those obtained in the usual manner. Where, however, the direct combustion of a steel, after mixing with red-lead, is practised, the use of an empty tube is recommended. It may be remarked, in parenthesis, that the direct combustion of steel borings with some oxidising reagent is essential for the accurate assay of samples containing large amounts of chromium and tungsten, such as are now made for taking deep cuts at high speeds; the results are low whenever a preliminary separation of the iron and carbon is made.

**Furnace Blanks.**—When air or oxygen is passed through a combustion furnace, the attached potash-bulb or soda-lime tube nearly always weighs more (or less) after the operation than it did before. This difference is not due, as is often supposed, to an imperfection in the furnace-tube or an impurity in the asbestos which accompanies the separated carbon. It is due almost entirely to an unequal desiccation of the gases as they enter and leave the potash-bulb. The question, therefore, of whether the calcium chloride prolong can completely dehydrate the gases is not very important; its efficiency depends on its accurate adjustment to the desiccating arrangements which stand before it in the train of purifiers. This adjustment is best attained by drying the material as bought in a uniform

manner. Even then perfection is not attained, and a blank determination is essential, and must be repeated as the calcium chloride becomes visually moist along the length of the prolong.

**Gravimetric Estimation of Sulphur.**—The most troublesome part of this process is the evaporation of the nitrohydrochloric acid solution of the steel to dryness. The tendency to spirt is very small until the clear dark solution shows signs of turbidity; up to this point, therefore, half a plate-full of samples may be safely boiled at a brisk rate. It is then most expedient to pass a rubber or leather strap round the uncovered beaker, and whirl it over a strong Bunsen flame. A hissing noise is heard until the mass becomes so thick that it can hardly move, and then a decided crackling may be noticed. When this is over, i.e., in about two minutes, the beaker may be placed on the hottest part of the plate without fear of spirting; but as the basic nitrates have dried partly up the sides of the beaker, it is better to replace the cover and allow it to stand in a less heated position so that the acid fumes may condense and run down the sides of the beaker. In this way the material is neatly collected on the bottom, and can be most effectively baked.

## ON ANTIMONY PENTIODIDE.

By R. W. EMERSON MACIVOR, F.I.C., &c.,  
Formerly Assistant Professor of Chemistry, Anderson's College  
(Medical Faculty), Glasgow.

In a paper read before the Chemical Society of London many years ago (*Chem. Soc. Journ.*, 1876, vol. i., p. 329) I contradicted the statement previously made by van der Eapt (*Arch. Pharm.*, [2], cxvii., p. 115), that antimony pentiodide is formed when a mixture of the elements in the required proportions is carefully heated, and also showed that it is not produced when antimony tri-iodide and iodine are brought together (with or without heat), either in the solid form or in solution in carbon disulphide; indeed, the results of my investigation led me to conclude that the pentiodide does not exist.

Pendleton, however, states (*CHEMICAL NEWS*, vol. xlviii., p. 97) that he succeeded in forming the compound by fusing antimony with an excess of iodine in an atmosphere of inert gas in a sealed tube kept at a temperature above the fusing-point of the mixture for an hour or two, the excess of iodine being subsequently removed by heating the material at a temperature not exceeding 130° C. The final product was found to have a composition corresponding "pretty closely" with SbI<sub>5</sub>, and "consisted of a homogeneous mass of crystalline structure and dark brown colour, distinctly different from the tri-iodide, decomposable by water or on prolonged exposure to moist air." The melting-point of the substance is given as being between 78—79°, notably lower than that of either the tri-iodide or iodine.

I made a quantity of the supposed SbI<sub>5</sub> in the manner described by Pendleton, conducting the operation in an atmosphere of pure dry carbon dioxide, and submitted it to careful examination, with the result that, however its low melting-point may be explained, I have formed the opinion that it consists of an intimate mixture of the tri-iodide and iodine.

On analysis (the iodine being determined by means of sodium thiosulphate) it was found to contain 80.153 per cent of iodine (126.9), theory for SbI<sub>5</sub> requiring 84.151; but after continuous heating for twelve hours at 125—130° in a slow current of dry carbon dioxide, this fell to 76.373 per cent; theory for SbI<sub>3</sub> being 76.032. It dissolved in hot carbon disulphide, yielding a solution possessing the characteristic colour imparted to that liquid by free iodine, and which, on cooling, deposited crystals of SbI<sub>3</sub> melting at 165°. By means of chloroform, in which the tri-iodide is only slightly soluble, I succeeded in completely dissolving out the free iodine from the substance,

and obtained pure  $\text{SbI}_3$ . On being decomposed by water the material yielded yellow oxyiodide, hydriodic acid, and much free iodine—a result to be expected from such a mixture as I believe Pendleton's product to be.

The Laboratory, 29, Fenchurch Street, E.C.

## NEW METHOD FOR PURIFYING POTABLE WATERS.

By P. GUICHARD.

A few years ago (*Bull. Soc. Chim.*, Series 3, vol. xix., p. 588) I read, before the Société Chimique, a paper on the action of metals and of organic matter on the permanganates. From the experiments described in this paper, I devised a method for the purification of potable waters, which consists of acting on the waters with permanganate of lime in excess for a sufficient time, and then destroying the excess of permanganate with iron; it is then only necessary to filter to separate the insoluble ferric and manganic oxides.

This filtration was the difficult part of the operation; at first I used a filter of absorbent cotton-wool, but the difficulty of arranging this in a convenient and practical manner caused me to abandon it, and to use instead a filter-press containing one or two sheets of sterilised paper. The filtration is very simple then, only it is necessary to change the paper more often than the cotton-wool; however, this changing is very easy and gives no trouble. While the cotton-wool lasted for several months, the paper must be changed once a week. Under these conditions we obtain a perfectly clear liquid, but if we continue filtering, we observe, after about twelve days, according to the amount of water consumed, that the filtered water, which is colourless at first, becomes yellow after a few minutes, and then gradually becomes more and more cloudy, and deposits ferric oxide.

Why does the water, which has kept clear for several days, suddenly become cloudy? For an explanation, it is necessary to examine into the phenomena closely. On leaving the vessel containing the iron, all the iron oxide is in the ferrous state, and is in great excess; this oxide combines with the carbonic acid in the water, forming ferrous carbonate, which is then dissolved in the excess of carbonic acid, but the ferrous oxide saturates this carbonic acid, and nothing remains in the water but neutral ferrous carbonate. Thus we have a very dilute solution of ferrous carbonate, since the carbonate is not at all easily soluble; this solution then passes into the filter. If, in the first place, we decant a portion of the clear solution, we observe that it becomes cloudy after a few moments, and deposits ferric oxide; if, on the other hand, we filter a small quantity, we obtain a clear liquid which does not become cloudy.

I believe that during filtration the cellulose molecule decomposes the ferrous carbonate, fixes the oxide of iron, becomes a mordant, and the water passes free from iron; so long as the cellulose is not saturated with iron, the water passes clear and remains limpid; but when the cellulose becomes saturated with the mordant, the water first passes clear and then becomes cloudy rapidly. Analogous facts in the chemical action of filter-papers have been observed before. Further, through the saturation of the free carbonic acid, the carbonate of lime is precipitated.

In my 1898 paper I endeavoured to reduce the permanganate by means of other metals than iron; nearly all metals reduce the permanganates, but none are so rapid in their action as iron, from which it follows that only iron can effect a reduction sufficiently rapid to give continuous purification. Still, in certain cases, other metals can be employed with advantage if the surface is increased to a sufficient extent to augment the rapidity of the action. I have used sheets of pure tin-foil with success. The

decolouration of permanganated water by tin takes place about twenty minutes after contact. This is rather a long time, but under certain circumstances, when only a little water is required for example, or when travelling, this is no drawback, as the apparatus is more simple and easy to use.

It suffices to fill a water-bottle with clean sterilised tin-foil, and to pour the permanganated water on it. The water-bottle is then attached to the filter, and the whole reversed over another water-bottle. The filtration is very rapid and easy on account of the absence of iron; nothing remains on the filter except a little manganic oxide. The simplicity of the apparatus and the rapidity of filtration compensate for the slight increase of time necessary for the decolouration of the permanganate.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 18.

## THE ESTIMATION OF BROMIC ACID BY THE DIRECT ACTION OF ARSENIOS ACID.\*

By F. A. GOOCH and J. C. BLAKE.

(Concluded from p. 217).

PORTIONS of this bromate were taken for reduction by arsenious acid. Solutions not exceeding 200 c.m.<sup>3</sup> in volume, containing the bromate and a considerable excess of arsenious oxide acidified with sulphuric acid of half-strength, were boiled for periods varying from ten to forty-five minutes, neutralised with potassium acid carbonate, and titrated with iodine. The results are shown in Table III.

TABLE III.

| KBrO <sub>3</sub> taken. | As <sub>2</sub> O <sub>3</sub> taken. | H <sub>2</sub> SO <sub>4</sub> (1:1). | Time. | As <sub>2</sub> O <sub>3</sub> unchanged. | As <sub>2</sub> O <sub>3</sub> oxidised. | Error, in terms of KBrO <sub>3</sub> . |
|--------------------------|---------------------------------------|---------------------------------------|-------|-------------------------------------------|------------------------------------------|----------------------------------------|
| Grm.                     | Grm.                                  | C.m.s.                                | Mins. | Grm.                                      | Grm.                                     | Grm.                                   |
| 1. 0.0701                | 0.1881                                | 5                                     | 10    | 0.0661                                    | 0.1220                                   | 0.0014—                                |
| 2. 0.0701                | 0.1881                                | 5                                     | 10    | 0.0650                                    | 0.1231                                   | 0.0009—                                |
| 3. 0.0701                | 0.2475                                | 5                                     | 10    | 0.1232                                    | 0.1243                                   | 0.0002—                                |
| 4. 0.0701                | 0.2475                                | 5                                     | 10    | 0.1236                                    | 0.1239                                   | 0.0004—                                |
| 5. 0.0701                | 0.2475                                | 5                                     | 25    | 0.1234                                    | 0.1241                                   | 0.0003—                                |
| 6. 0.0701                | 0.2475                                | 5                                     | 25    | 0.1234                                    | 0.1241                                   | 0.0003—                                |
| 7. 0.1402                | 0.4950                                | 3                                     | 15    | 0.2479                                    | 0.2471                                   | 0.0012—                                |
| 8. 0.1402                | 0.4950                                | 3                                     | 15    | 0.2476                                    | 0.2474                                   | 0.0010—                                |
| 9. 0.1400                | 0.6188                                | 7                                     | 20    | 0.3708                                    | 0.2480                                   | 0.0004—                                |
| 10. 0.1400               | 0.6188                                | 7                                     | 20    | 0.3710                                    | 0.2478                                   | 0.0005—                                |
| 11. 0.1400               | 0.6188                                | 7                                     | 20    | 0.3706                                    | 0.2482                                   | 0.0003—                                |
| 12. 0.1400               | 0.6188                                | 7                                     | 30    | 0.3708                                    | 0.2480                                   | 0.0004—                                |
| 13. 0.1400               | 0.6188                                | 7                                     | 45    | 0.3711                                    | 0.2477                                   | 0.0006—                                |

Here again, as in the experiments of Table I., the indications of the process of reduction of the bromate by arsenious acid point to a slight deficiency in the oxidising power of the bromate. The mean deficiency, 0.0006 gram., differs from the indications of the process of reduction of the same sample by the potassium-iodide method by about 0.0003+ gram.

The question now arises as to what is the impurity in the bromate. In a product re-crystallised several times, and in one in which no chloride can be detected, as was the case with the bromate of these experiments, the impurity most natural to look for is potassium chlorate, which might resist removal in the process of purification by re-crystallisation. The preparation of bromate upon which these last experiments were made was therefore tested by igniting it and treating the residue with potassium bichromate and sulphuric acid, volatilising and collecting any chloro-chromic anhydride thus formed, and converting the last into lead chromate (Goode and Brooks, *Am. Journ. Sci.*, 1890, xl., p. 287). Traces of chlorine were thus found, which, not appearing in a similar test upon the unignited bromate, must have had their origin in

\* Contribution from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, [4], xiv., No. 82.

chlorate intercrystallised with the bromate. In a former paper from this laboratory (Gooch and Smith, *Am. Journ. Sci.*, 1891, xlii., p. 220) it has been shown that a chlorate may be determined by adding to it in solution potassium iodide in known amount and an excess of an arseniate and sulphuric acid, boiling the mixture between definite limits of concentration, determining by titration with iodine the amount of arsenious oxide produced, and calculating the amount of chlorate present, from the difference between the amount of arsenious oxide thus produced and that which should be produced if the whole amount of iodide added were allowed to act upon the arseniate alone. There appears to be no reason why a bromate treated by this process should not leave a similar record of its oxidising power. A mixture of bromate and chlorate should, therefore, in this process, leave a record of the full amount of oxidation of which both together are capable. The following table contains the account of experiments made in this manner upon the sample of bromate, the action of which in the iodide method and in the arsenious acid method is recorded in Tables II. and III.

TABLE IV.

H<sub>2</sub>SO<sub>4</sub> (1:1), 20 c.m.<sup>3</sup>; Initial Volume, 105 to 170 c.m.<sup>3</sup>; Final Volume, 35 c.m.<sup>3</sup>.

| KBrO <sub>3</sub> taken. | H <sub>2</sub> KAsO <sub>4</sub> taken. | I value of KI taken. | Iodine corresponding to As <sub>2</sub> O <sub>3</sub> produced. | Iodine corresponding to KBrO <sub>3</sub> . | Error in terms of KBrO <sub>3</sub> . |
|--------------------------|-----------------------------------------|----------------------|------------------------------------------------------------------|---------------------------------------------|---------------------------------------|
| Grm.                     | Grms.                                   | Grm.                 | Grm.                                                             | Grm.                                        | Grm.                                  |
| 1. 0.0700                | 2                                       | 0.4146               | 0.0948                                                           | 0.3198                                      | 0.0002+                               |
| 2. 0.0700                | 2                                       | 0.4146               | 0.0954                                                           | 0.3192                                      | 0.0000                                |
| 3. 0.0700                | 2                                       | 0.4146               | 0.0969                                                           | 0.3177                                      | 0.0003-                               |
| 4. 0.0700                | 2                                       | 0.4146               | 0.0975                                                           | 0.3171                                      | 0.0004-                               |
| 5. 0.1400                | 2                                       | 0.7832               | 0.1458                                                           | 0.6374                                      | 0.0001-                               |
| 6. 0.1400                | 2                                       | 0.7832               | 0.1463                                                           | 0.6369                                      | 0.0002-                               |
| 7. 0.1400                | 2                                       | 0.7832               | 0.1462                                                           | 0.6370                                      | 0.0002-                               |

The mean error of these determinations, in which all oxidising material is calculated as bromate, is not far from 0.0001- gm.

TABLE V.

A. Arsenious Acid Method.  
Volume not exceeding 100 c.m.<sup>3</sup>.

| KBrO <sub>3</sub> taken.           | As <sub>2</sub> O <sub>3</sub> taken. | H <sub>2</sub> SO <sub>4</sub> (1:1). | Time. | As <sub>2</sub> O <sub>3</sub> unchanged. | As <sub>2</sub> O <sub>3</sub> oxidised. | Error in terms of KBrO <sub>3</sub> . |
|------------------------------------|---------------------------------------|---------------------------------------|-------|-------------------------------------------|------------------------------------------|---------------------------------------|
| Grm.                               | Grm.                                  | Grms.                                 | Mins. | Grm.                                      | Grm.                                     | Grm.                                  |
| <i>At the Boiling Temperature.</i> |                                       |                                       |       |                                           |                                          |                                       |
| 0.0704                             | 0.2475                                | 5                                     | 10    | 0.1241                                    | 0.1234                                   | 0.0010-                               |
| 0.0704                             | 0.2475                                | 5                                     | 15    | 0.1239                                    | 0.1236                                   | 0.0009-                               |
| 0.0704                             | 0.2475                                | 5                                     | 15    | 0.1236                                    | 0.1239                                   | 0.0007-                               |
| <i>On the Steam-bath, -80°.</i>    |                                       |                                       |       |                                           |                                          |                                       |
| 0.0704                             | 0.2475                                | 5                                     | 30    | 0.1241                                    | 0.1229                                   | 0.0013-                               |
| 0.0704                             | 0.2475                                | 5                                     | 30    | 0.1246                                    | 0.1234                                   | 0.0010-                               |
| <i>At Atmospheric Temperature.</i> |                                       |                                       |       |                                           |                                          |                                       |
| 0.0704                             | 0.2475                                | 5                                     | 10    | 0.2066                                    | 0.0409                                   | 0.0474-                               |
| 0.0704                             | 0.2475                                | 5                                     | 10    | 0.1991                                    | 0.0488                                   | 0.0426-                               |
| 0.0704                             | 0.2475                                | 5                                     | 30    | 0.1533                                    | 0.0922                                   | 0.0185-                               |
| 0.0704                             | 0.2475                                | 5                                     | 60    | 0.1231                                    | 0.1244                                   | 0.0004-                               |
| 0.0704                             | 0.2475                                | 5                                     | 120   | 0.1242                                    | 0.1233                                   | 0.0011-                               |
| 0.0704                             | 0.2475                                | 5                                     | 120   | 0.1238                                    | 0.1237                                   | 0.0009-                               |

B. Arseniate-Iodide Method.

H<sub>2</sub>SO<sub>4</sub> (1:1), 20 c.m.<sup>3</sup>; Initial Volume, 110 c.m.<sup>3</sup>; Final Volume, 35 c.m.<sup>3</sup>.

| KBrO <sub>3</sub> taken. | H <sub>2</sub> KAsO <sub>4</sub> taken. | I value of KI taken. | Iodine corresponding to As <sub>2</sub> O <sub>3</sub> produced. | Iodine corresponding to KBrO <sub>3</sub> . | Error in terms of KBrO <sub>3</sub> . |
|--------------------------|-----------------------------------------|----------------------|------------------------------------------------------------------|---------------------------------------------|---------------------------------------|
| Grm.                     | Grms.                                   | Grm.                 | Grm.                                                             | Grm.                                        | Grm.                                  |
| 0.0704                   | 2                                       | 0.3812               | 0.0607                                                           | 0.3205                                      | 0.0001-                               |
| 0.0704                   | 2                                       | 0.3812               | 0.0588                                                           | 0.3224                                      | 0.0003+                               |
| 0.0704                   | 2                                       | 0.3812               | 0.0595                                                           | 0.3217                                      | 0.0001+                               |
| 0.0704                   | 2                                       | 0.3812               | 0.0590                                                           | 0.3222                                      | 0.0002+                               |
| 0.0704                   | 2                                       | 0.3812               | 0.0615                                                           | 0.3197                                      | 0.0003-                               |
| 0.0647                   | 2                                       | 0.3812               | 0.0839                                                           | 0.2973                                      | 0.0004+                               |

In the case of still another preparation of potassium bromate, made by acting with commercial bromine on potassium hydroxide, crystallising and re-crystallising several times, determinations by means of arsenious acid, and by the arseniate-iodide process, resulted as shown in Table V.

The mean error of the arseniate-iodide process applied to this particular sample of bromate is 0.0001+ gm. in terms of bromate; the arsenious acid method shows a mean deficiency of about 0.0009 gm. for the smaller amount of bromate employed, if, as is obviously reasonable, those results are omitted from the averages which were obtained by standing at the ordinary temperature for periods less than one hour.

It appears, therefore, that the deficiency in the indications of the iodide process and the arsenious acid process are satisfactorily accounted for by the presence in the bromate of traces of chlorate; and that the oxidising power of a bromate may be determined by boiling it in solution with a known excess of arsenious oxide and an excess of sulphuric acid, and determining the amount of arsenious oxide remaining unchanged. A chlorate, as we have found by direct experiment, is scarcely affected by this treatment.

## OUR PRESENT KNOWLEDGE OF AROMATIC DIAZO-COMPOUNDS.\*

By GILBERT THOMAS MORGAN, D.Sc., F.I.C.

(Concluded from p. 216).

### D. The Diazonium Radicle compared with Quaternary Ammonium Ions.

THE change of opinion in favour of the Strecker-Blomstrand formula for the diazonium salts was in the first place based on certain general analogies existing between the salts of nitrogenous bases.

The older view of the constitution of diazonium salts indicates that these substances form an exception to the rule that basic nitrogen is pentavalent in its salts, and yet the compounds in question behave as the salts of bases, more powerful than the aromatic amines from which they are derived. The diazonium bases are capable of combining with the weaker acids, and yield soluble alkaline carbonates, e.g., (C<sub>6</sub>H<sub>5</sub>N<sub>2</sub>)<sub>2</sub>CO<sub>3</sub>, and unstable nitrites and acetates. Moreover, this view involves the assumption that the compound C<sub>6</sub>H<sub>5</sub>N:NOH can react as a strong ammonium base towards acids and as a distinctly acidic substance towards the alkali hydroxides. Hantzsch showed that the diazonium salts of the mineral acids are strongly ionised in dilute solution, but do not exhibit any trace of hydrolytic dissociation; the ionisation is, however, considerably diminished by the introduction of negative radicles into the aromatic nucleus of the diazonium compound (*Ber.*, 1895, xxviii., 1734).

Determinations of the electrical conductivity of solutions of benzene-diazonium chloride and nitrate showed that the benzenediazonium radicle is strictly comparable with other quaternary ammonium ions. The rate of migration of the benzenediazonium ion at 25° is 45.7, the corresponding constants for the methylpyridinium, C<sub>5</sub>H<sub>5</sub>NCH<sub>3</sub>, and tetramethylammonium ions, N(CH<sub>3</sub>)<sub>4</sub>, being 44.3 and 43.6 respectively.

The molecular conductivity of the solutions of diazonium salts increases with the dilution just as in the case of the corresponding potassium and ammonium compounds.

A physico-chemical study of the solutions of benzene-diazonium hydroxide showed that the affinity constant of this base at 0° is seventy times greater than that of ammonium hydroxide, and somewhat exceeds that of piperidine. The affinity constants of methoxybenzene-

\* Report presented to Section B, British Association, Belfast Meeting, 1902.

diazonium hydroxide and  $\psi$ -cumidinediazonium hydroxide are even greater, and approximate closely to those of the alkali hydroxides. The effect of introducing halogen radicles into the aromatic nucleus is indicated in a striking manner in the following table:—

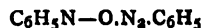
|                                                                  | $k$ =velocity constant. |
|------------------------------------------------------------------|-------------------------|
| $\text{C}_6\text{H}_5\text{N}_2\text{OH}$ .. ..                  | 0.123                   |
| $\text{BrC}_6\text{H}_4\text{N}_2\text{OH}$ .. ..                | 0.0149                  |
| $2:4\text{-Br}_2\text{C}_6\text{H}_3\text{N}_2\text{OH}$ .. ..   | 0.0136                  |
| $2:4:6\text{-Br}_3\text{C}_6\text{H}_2\text{N}_2\text{OH}$ .. .. | 0.0014                  |

A comparison of the electrical conductivity experiments with the results obtained in the hydrolysis of ethyl acetate by benzenediazonium hydroxide shows that, in 1/128 N solutions at  $0^\circ$ , approximately 33 per cent of the base exists in the ionised condition.

The ionisation detected in the hydrolysis experiment is greater than that indicated by the conductivity determinations, and this shows that the electrolytic dissociation is exclusively due to the reaction—



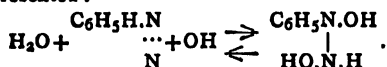
and not to the existence of a diazonium *syn*-diazo-oxide,—



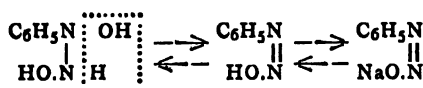
**Condition of the Non-ionised Diazonium Hydroxide.**—The solution of benzenediazonium hydroxide differs essentially from one containing piperidine in its behaviour towards alkali hydroxides, generating with these reagents an appreciable amount of heat exactly like a weak acid. This reaction is also indicated by determinations of the electrical conductivities of solutions of the diazonium hydroxide when treated with one, two, or molecular proportions of sodium hydroxide.

The simplest explanation of this phenomenon is based on the assumption that the non-ionised portion of the diazonium hydroxide exists in solution in a hydrated form which, on the addition of alkali hydroxide, yields the alkali salt, the acidic *syn*-diazo-hydroxide behaving in this respect precisely like the similarly constituted oximes.

Before the addition of alkali the equilibrium may be thus represented:—

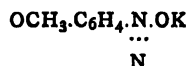


The addition of the alkali hydroxide causes the hydrate to lose water, forming the *syn*-diazo-hydroxide, which then gives rise to a certain amount of sodium derivative,—



(Hantzsch and Davidson, *Ber.*, 1898, xxxi., 1612).

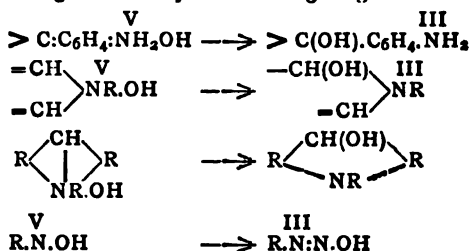
If this hypothesis is not accepted, then it must be assumed that a quaternary ammonium hydroxide, having the functions of a strong base, is also capable of behaving as an acid, and yielding an alkali salt. The diazonium hydroxide derived from anisidine, for example, is a base comparable in strength with the alkalis, and yet it gives rise to a stable potassium derivative (*Ber.*, 1900, xxxiii., 2158), which, according to the alternative theory, has the formula—



There are, however, absolutely no other examples of strong alkalis behaving in this way.

On the other hand, the hypothesis based on the change of configuration due to the labile nature of the diazonium hydroxides, brings these bases into line with other quaternary ammonium derivatives. It is well known that almost all the quaternary ammonium hydroxides, excepting

those in which the nitrogen is attached to four fully saturated hydrocarbon radicles, are unstable, and pass into substances in which the hydroxyl group is no longer attached to the aminic nitrogen. This alteration is noticed in the bases of the rosaniline, pyridine, and acridine series; the transformations taking place in each case being indicated by the following diagrams:—

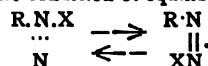


Ammonium derivatives.

Pseudo-ammonium derivatives.

According to this generalisation the *syn*-diazo-hydroxides are to be regarded as pseudo-diazonium derivatives (*Ber.*, 1899, xxxii., 3109; 1900, xxxiii., 278).

**Solid Diazonium Halides.**—The unstable character of certain diazonium halides seems to be connected in some way with their colour, the most highly coloured salts being very explosive. These coloured salts are found to give colourless solutions, in which the salt is undoubtedly in the form of a diazonium compound. The appearance of colour in the solid state is considered to be due to the formation of a certain amount of *syn*-diazo-derivative, in accordance with the condition of equilibrium:—

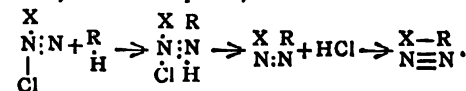


The amount of coloured *syn*-compound present depends largely on the nature of the acid radicle X.

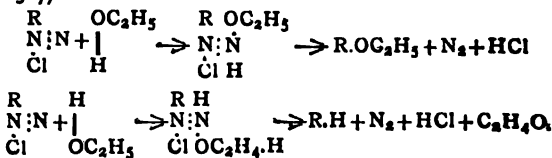
The diazonium chlorides, and nearly all the bromides, are colourless and comparatively stable. The iodides are all coloured, and very explosive, the thiocyanates are somewhat less coloured than the iodides, and are intermediate in stability between these and the bromides.

The influence of substituent radicles in the aromatic radicle R is scarcely noticeable with the diazonium chlorides; but with the other salts it is found that the stability is increased by the introduction of methyl and methoxy groups, and diminished by that of acidic radicles (*Ber.*, 1900, xxxiii., 2179). At low temperatures even the very explosive diazonium salts become stable, and it is noticed that, on cooling, their colour is perceptibly diminished (*Ber.*, 1901, xxxiv., 4166).

**The Residual Affinity of Diazonium Salts** (*Ber.*, 1897, xxx., 2548; 1898, xxxi., 2053).—The characteristic fission of diazonium salts is very probably due to the preliminary addition of a reagent HR, and the subsequent elimination of the hydride of the acid radicle with the formation of an unstable *syn*-diazo-compound,—



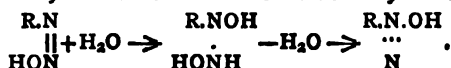
This hypothesis affords a general explanation of the Sandmeyer reaction, and also of the interaction between diazonium salts and water or alcohol (*Ber.*, 1900, xxxiii., 2517).



The cuprous salts of the Sandmeyer reaction combine with *syn*-diazo-compounds to form coloured double salts of the azo-type, and, in this way, induce the conversion of the diazonium salt into the pseudo-diazonium or *syn*-form which subsequently decomposes in the characteristic manner.

The diazonium perhalides are substances analogous with the periodides of potassium, caesium, and the quaternary ammonium bases. The halogen atoms are certainly not attached to both nitrogen atoms, since the compound  $C_6H_5N_2ClBrI$  is produced either from  $C_6H_5N_2Cl$  and  $IBr$  or from  $C_6H_5N_2Br + ICl$  (Ber., 1895, xxviii., 2754).

**Hydrolytic Dissociation of the Alkali Diazo-oxides.**—It has already been seen that the metallic *syn*- and *anti*-diazo-oxides are much alike in their general chemical behaviour, providing that their reactions are studied in fairly alkaline solutions. The *syn*-isomerides, however, exhibit anomalous properties in dilute neutral solution, and this phenomenon has been found, by determinations of the electrical conductivity (Ber., 1898, xxxi., 1612), to be due to the abnormally large hydrolytic dissociation of these substances. This is explained by the hypothesis already employed in considering the condition of the dissolved diazonium hydroxide. The *syn*-diazo-oxide exhibits a higher degree of hydrolytic dissociation than potassium cyanide or other dissociating salts, because the acid set free by hydrolysis is not stable, but passes into the intermediate hydrate and thence to the diazonium hydroxide:—



**Anti-diazo-oxides and Primary Nitrosamines.**—The alkali diazo-oxides behave like the salts of a distinctly acid substance, and are not greatly hydrolysed in dilute solution. When the dissolved salt is treated with the equivalent amount of hydrochloric acid, the electrical conductivity of the resulting solution shows that the only electrolyte present is the alkali chloride produced by the reaction. The solution obtained is perfectly neutral, and conversely when the substance which is set free by the acid is treated with the equivalent quantity of alkali the product is neutral. These phenomena indicate that the substance set free from the *anti*-diazo-oxide belongs to the class of pseudo-acids (Ber., 1899, xxxii., 575, 1703), and accordingly the product is best represented as being a primary nitrosamine,—



Anti-diazo-oxide.      Labile.      Stable.

The nitrosamines—



have the characters of pseudo-acids; they do not react with phosphorus penta-chloride, acetyl chloride, or ammonia in indifferent solvents, and dissolve only gradually in acids regenerating the diazonium salt.

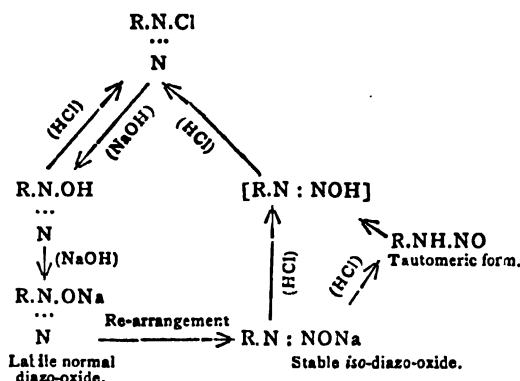
**Summary.**—The theory of diazo-compounds, which ignores stereo-chemical differences and assumes that the diazo-oxides are structurally dissimilar, may be summarised in Diagram A.

The stereo-chemical theory may be briefly outlined as in Diagram B.

The substances indicated in square brackets are hypothetical intermediate compounds, the existence of which is assumed in order to explain the tautomeric changes.

In our present state of knowledge a choice still remains between a theory of diazo-compounds which, although not assuming any spatial relationships, nevertheless offers an explanation of the isomerism of these substances which is applicable to this series of nitrogen-derivatives

DIAGRAM A.



alone, and a stereo-chemical hypothesis which correlates the diazo-derivatives with other nitrogen compounds of a similar type, and affords a general interpretation of a considerable group of experimental facts.

The assumption of *syn*- and *anti*-isomerism explains the production of the two series of coloured cyanides and sulphonates from the colourless diazonium salts. Moreover, the stereo-chemical hypothesis renders it possible to connect the relationship between the diazonium and *syn*-diazo-hydroxides with the general theory of quaternary ammonium compounds. In a similar manner the behaviour of the primary aromatic nitrosamines and the *anti*-diazo-oxides may be correlated with the general theory of pseudo-acids and their salts.

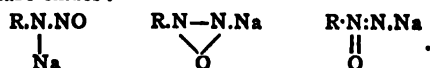
It may be urged against the stereo-chemical theory of diazo-compounds that the *syn*-diazo series is still very defective, being confined to three classes of substances, the metallic diazo-oxides, the diazo-cyanides, and diazo-sulphonates, and it is still possible that the isomeric cyanides and sulphonates may be shown to be structurally different. On the other hand, the view that the *syn*-diazo-compounds have a diazonium configuration leads to conclusions which are not in accordance with experimental facts. This is particularly the case with the metallic diazo-oxides, for it becomes necessary to assume that the strongly basic diazonium hydroxides also react as acids. This hypothesis is in the highest degree improbable, there being no other example of a quaternary ammonium hydroxide possessing this dual nature.

#### Addendum.

**I. Additional Notes on the Stereochemical Theory.**—Since writing the foregoing report the author has received a communication from Professor Hantzsch containing the latest developments of the stereochemistry of the diazo-compounds.

The alternative views are fully discussed, and the plane formulae suggested for the *syn*-diazo derivatives, on the assumption that these substances differ structurally from their *anti*-isomerides, are shown to be incapable of accounting for the observed facts.

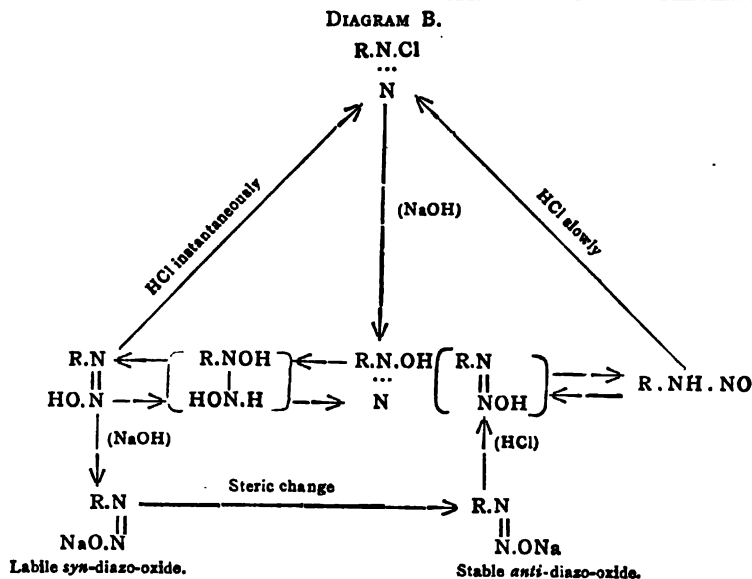
In addition to the diazonium formula, three other structural formulae have been advanced for the metallic *syn*-diazo-oxides:—



These configurations fail, in the following important points, to account for the behaviour of these metallic diazo-derivatives:—

1. They do not furnish a simple explanation of the reciprocal transformation of these substances into diazonium salts and *vice versa*.

2. They do not indicate that capacity for coupling with



phenols to form azo compounds which is so essentially characteristic of the *syn*-diazoo series.

3. Moreover, they involve the additional assumption that the isomerism of the metallic diazo-oxides differs essentially from that of the diazo-cyanides and diazo-sulphonates, inasmuch as the latter compounds cannot be formulated on these lines. This hypothesis is, however, unjustifiable, for the *syn*-diazoo derivatives of the three series (oxides, cyanides, and sulphonates) have comparable properties.

The only other structurally dissimilar formulæ available for the *syn*-diazoo-cyanides and diazo-sulphonates are the diazonium formula—

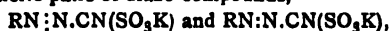


N

and the configuration  $\text{R.N} : \text{N.CN(SO}_3\text{K)}$ .

The former of these is not in accordance with the general properties (colour and sparing solubility) of these derivatives, and, moreover, the electrolytic dissociation of the diazo-sulphonates into two ions confirms the view that the compounds are diazo-sulphonates,  $\text{R.N:N.SO}_3\text{K}$ , and not diazonium sulphites.

The second formula involves an entirely new assumption—namely, that of isomerism due to change of valency; but even if this possibility be granted, the corresponding formulation does not furnish an explanation of the close relationship existing between the *syn* and *anti* series, for the isomeric pairs of diazo-compounds,—



being derivatives of pentavalent and trivalent nitrogen respectively, ought to differ completely in their chemical reactions.

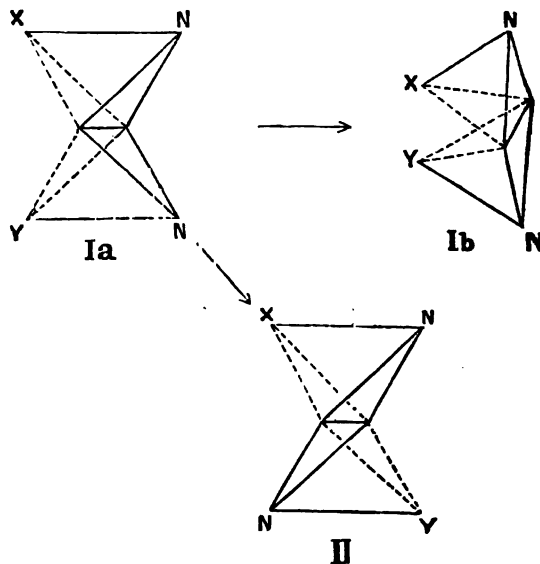
In developing the stereochemical theory of the diazo-derivatives, Hantzsch adopts the space formulæ employed in explaining the isomerism of the oximes.

The assumption involved is that the valencies of the trivalent nitrogen atom are directed along the convergent edges of a regular tetrahedron, the atom itself being situated at the apex of the solid angle thus produced.

When two nitrogen atoms become doubly linked in a molecule, the compound which results is capable of existing in two stereomeric forms corresponding with the diagrams Ia and II.

By means of these diagrams the *syn*- and *anti*-diazoo derivatives may be compared with the stereomeric oximes and the *cis*- and *trans*-isomerides of doubly-linked carbon compounds.

The great instability of the *syn*-diazoo series may be explained in terms of v. Baeyer's "tension" theory. According to this hypothesis, the valencies linking together two conjugated atoms tend to set themselves in the same straight line, and when this is not possible, a stress is produced in the molecule which renders it unstable and prone to undergo re-arrangement.

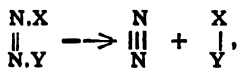


The diagram Ia indicates a molecule in this unstable condition, the linking valencies of the two nitrogen atoms being inclined at an angle to each other. This intramolecular strain can be relieved by placing the sides of the tetrahedra bounded by continuous lines in the same plane, the necessary change in the spatial relationship of these figures being produced either by re-arrangement (Ia to II), or more simply by rotating the tetrahedra about their common edge (Ia and Ib).

The former operation represents the transformation of the labile *syn*-derivative into its stable *anti* isomeride; the latter indicates the final condition of the *syn*-com-



pound itself, and furnishes an explanation of the typical diazo-fission,—



brought about by the close proximity of the radicles attached to the diazo-complex.

II. *Isolation of the Anti-diazohydroxides* R.N:N.OH.—Hantzsch has recently discovered that in certain cases both forms of the anti-diazohydroxides are capable of separate existence. The nitrosamine forms have already been studied; they are comparatively stable yellow compounds belonging to the category of pseudo-acids, and yield negative results when treated with phosphorus pentachloride, acetyl-chloride, phenyl-carbimide, or a dry ethereal solution of ammonia.

The newly isolated oxime forms—



corresponding with the metallic anti-derivatives R.N:NONa(K), are labile colourless substances; they are true acids reacting with the agents enumerated in the preceding sentence.

The syn-diazohydroxides are only known in solution.

III. *Transformation of Diazonium Salts*.—The following observations should be added to the summary of miscellaneous substitutions:—

Meldola and Eyre have shown that in the dinitro-anisidines a nitro-group situated in the ortho- or para-position with respect to amidogen is replaced either by chlorine, when the base is diazotised in the presence of hydrochloric acid (Meldola and Eyre, *Trans.*, 1901, lxxix, 1077; 1902, lxxxi, 988).—

$\text{OMe.C}_6\text{H}_3(\text{NO}_2)_2\text{N}_2\text{Cl} \rightarrow \text{OMe.C}_6\text{H}_3\text{Cl}(\text{NO}_2)_2\text{N}_2\text{NO}_2$ , or by hydroxyl, when the operation is carried out in sulphuric acid.

The author has quite recently noticed a similar transformation in the case of *o*-nitro  $\beta$ -naphthylamine when diazotised in the presence of hydrochloric acid,  $\text{NO}_2\text{C}_{10}\text{H}_6\text{N}_2\text{Cl} \rightarrow \text{ClC}_{10}\text{H}_6\text{N}_2\text{NO}_2$ , and further rearrangements of this nature have been observed by other investigators (Gaess and Ammelburg, *Ber.*, 1894, xxvii, 2211; Meldola and Streatfield, *Trans.*, 1895, lxxvii, 909; *Fr. Pat.* 315932, February 28, 1902).

Further under these conditions bromine may be replaced by chlorine, it does not appear possible to remove fluorine or iodine from the aromatic nucleus through the agency of the diazo-reaction (Hantzsch).

## A NEW SEPARATION OF THORIUM FROM CERIUM, LANTHANUM, AND DIDYMIUM, AND ITS APPLICATION TO THE ANALYSIS OF MONAZITE.\*

By FLOYD J. METZGER.

(Continued from p. 219).

### Method of Analysis.

MAKE the solution of the neutral nitrate 40 per cent alcohol (volume about 150 to 175 c.c. with 0.1 to 0.2 grm. thorium dioxide and about the same amount of admixed oxides), then add 12 to 15 c.c. of fumaric acid solution (about 0.10 grm. per 10 c.c.) for each 0.1 grm. thorium dioxide, and heat to boiling (75–80° C.); allow to stand a few minutes until the precipitate settles, then filter (while still hot) through a long-stem funnel, or by suction.

\* Contribution from the Havemeyer Laboratories, Columbia University. Read at the meeting of the New York Section of the American Chemical Society. From the *Journal of the American Chemical Society*, xlv., No. 10.

TABLE.

| CeO <sub>2</sub> . | La <sub>2</sub> O <sub>3</sub> . | Di <sub>2</sub> O <sub>3</sub> . | Fumaric |         | ThO <sub>2</sub> . |        |
|--------------------|----------------------------------|----------------------------------|---------|---------|--------------------|--------|
|                    |                                  |                                  | acid.   | Volume. | Taken.             | Found. |
| A.                 |                                  |                                  |         |         |                    |        |
| 0.2000             | —                                | —                                | 25      | 150     | 0.1725             | 0.1730 |
| 0.2000             | —                                | —                                | 25      | 150     | 0.1961             | 0.1982 |
| —                  | 0.2000                           | —                                | 25      | 150     | 0.1921             | 0.1941 |
| —                  | 0.2000                           | —                                | 25      | 150     | 0.1961             | 0.1990 |
| —                  | 0.2000                           | —                                | 25      | 150     | 0.1882             | 0.1907 |
| —                  | 0.2000                           | —                                | 25      | 150     | 0.1961             | 0.1978 |
| —                  | —                                | 0.2000                           | 25      | 150     | 0.1961             | 0.1992 |
| —                  | —                                | 0.2000                           | 25      | 150     | 0.1961             | 0.1972 |
| —                  | —                                | 0.2000                           | 25      | 150     | 0.1882             | 0.1901 |
| —                  | —                                | 0.2000                           | 25      | 150     | 0.1961             | 0.1979 |
| B.                 |                                  |                                  |         |         |                    |        |
| 0.2000             | —                                | —                                | 25      | 150     | 0.1961             | 0.1966 |
| 0.2000             | —                                | —                                | 25      | 150     | 0.1961             | 0.1957 |
| —                  | 0.2000                           | —                                | 25      | 150     | 0.1961             | 0.1964 |
| —                  | 0.2000                           | —                                | 25      | 150     | 0.1961             | 0.1959 |
| —                  | —                                | 0.2000                           | 25      | 150     | 0.1961             | 0.1965 |
| —                  | —                                | 0.2000                           | 25      | 150     | 0.1968             | 0.1965 |
| C.                 |                                  |                                  |         |         |                    |        |
| 0.283              | 0.133                            | 0.157                            | 10      | 175     | 0.0557             | 0.0558 |
| 0.283              | 0.133                            | 0.157                            | 25      | 200     | 0.0572             | 0.0571 |
| 0.283              | 0.133                            | 0.157                            | 10      | 175     | 0.0572             | 0.0574 |
| 0.283              | 0.133                            | 0.157                            | 10      | 175     | 0.0557             | 0.0558 |
| 0.566              | 0.266                            | 0.314                            | 15      | 350     | 0.1115             | 0.1114 |
| 0.566              | 0.266                            | 0.314                            | 15      | 350     | 0.1129             | 0.1132 |
| D.                 |                                  |                                  |         |         |                    |        |
| 0.1000             | 0.1000                           | 0.1000                           | 10      | 120     | 0.0995             | 0.0990 |
| 0.1000             | 0.1000                           | 0.1000                           | 10      | 120     | 0.1003             | 0.0995 |
| 0.1000             | 0.1000                           | 0.1000                           | 25      | 130     | 0.1529             | 0.1523 |
| 0.1000             | 0.1000                           | 0.1000                           | 40      | 150     | 0.2007             | 0.2002 |
| 0.1000             | 0.1000                           | 0.1000                           | 40      | 150     | 0.2015             | 0.2010 |
| 0.1000             | 0.1000                           | 0.1000                           | 60      | 150     | 0.3011             | 0.3005 |
| 0.1000             | 0.1000                           | 0.1000                           | 60      | 150     | 0.3019             | 0.3023 |
| 0.0500             | 0.0500                           | 0.0500                           | 25      | 200     | 0.1961             | 0.1963 |
| 0.0500             | 0.0500                           | 0.0500                           | 25      | 200     | 0.1968             | 0.1969 |
| 0.0250             | 0.0250                           | 0.0250                           | 25      | 200     | 0.1976             | 0.1971 |
| 0.0250             | 0.0250                           | 0.0250                           | 25      | 200     | 0.1991             | 0.1985 |

Wash the precipitate four or five times with hot 40 per cent alcohol, then put paper and precipitate into a platinum crucible, ignite, and weigh as thorium dioxide. A number of analyses were made in this way, using mixtures of thorium with cerium, lanthanum, and didymium, and it was found that the thorium invariably carried with it an appreciable quantity of the impurity which could not be washed out, nor could it be prevented by the addition of such salts as ammonium chloride, ammonium nitrate, &c.

A few results will show the extent to which these impurities are carried down (see Table, A).

Re-precipitation was next tried, and gave very satisfactory results. For this re-precipitation it is only necessary to dissolve the precipitate of thorium fumarate (formed by the first precipitation) off the filter in hot dilute hydrochloric acid—or better, place the paper and precipitate back into the beaker, add a little dilute hydrochloric acid, heat, then filter off the paper—and evaporate to dryness on a water-bath. During the process of this evaporation some of the salt adheres to the sides of the beaker, but this is easily obviated by shaking the beaker occasionally and washing down with a few drops of water from a wash-bottle.

When the residue is free from acid (better add a few cubic centimetres of water and evaporate a second time), add about 50 c.c. water (leaving the beaker on the water-bath) and stir the residue loose from the bottom with a rubber-capped stirring rod; some carbonaceous matter from the fumaric acid, partly decomposed, and also some fumaric acid and a trace of thorium fumarate, will remain insoluble at this point, but need not be regarded. Add

alcohol sufficient to make the solution 40 per cent, dilute to the proper volume with 40 per cent alcohol, add a little fumaric acid (8 to 10 c.c.), and precipitate as before; wash, ignite, and weigh.

Hydrochloric acid was used instead of nitric for the solution of the thorium fumarate, for the reason that nitric acid formed, on evaporation, a deposit which was practically insoluble in water. The accompanying results will show the accuracy of the separation (see Table, B).

Mixtures of thorium, cerium, lanthanum, and didymium were then made in the proportions in which they are usually found in monazite sand, and analysed the same as above, with the results shown (see Table, C).

Mixtures containing equal amounts of thorium, cerium, lanthanum, and didymium, and mixtures containing thorium in excess, were then analysed as shown (see Table, D).

(To be continued).

## A NEW LABORATORY-SHAKING MACHINE.

By GERALD T. MOODY.

THE machine described is a modification of an "American cream-drink shaker," is very efficient, is inexpensive, and does not get out of order. The glass bottles or other

double bracket on which the bottles, &c., rest, and at the upper end a double bracket carrying tightening screws. This framework and the vertical rod are given vertical motion by means of an iron connecting rod, at the upper end fastened to the working rod by a pin-joint; at the lower end given rotary motion by a crank-pin affixed to a small cast-iron balanced fly-wheel. The fly-wheel is attached by a set screw to a horizontal shaft supported by the iron standard, and carrying a cast-iron V-pulley about 3½ inches in diameter. Motion is transmitted to this pulley by means of an 1½-inch grooved hand-wheel and round leather belt, the hand-wheel running on a fixed horizontal shaft, bolted to the standard, and having vertical adjustment.

To use the machine for shaking, the bottle or other containing vessel is placed on a felt pad on one of the arms of the moving frame, and held down by the tightening screw, between which and the bottle-stopper is placed an indiarubber bung.

At the Central Technical College, where the machine is run by power, a 3" × 1" inch cast-iron pulley is bolted to the hand-wheel, and a similar pulley runs loose on the fixed horizontal shaft, which is considerably longer than the one used with the hand-wheel alone. A 1 inch single strap leather belt transmits power from a 2½-inch gas-engine shaft running at 200 revolutions. The machine then runs at nine complete oscillations per second, which



FIG. 1.—Front View.

vessels, the contents of which are to be shaken, are gripped in a frame and given a rapid vertical oscillatory motion.

The machine consists of a cast-iron standard (Figs. 1 and 2), which carries two bbred parallel brackets 3 inches apart, and through these passes a vertical working rod. To the top of the rod is attached an iron framework, comprising a central stem to which are fixed, at the lower end, a cast



FIG. 2.—Back View.

is very effectual in working. The belt is struck from loose to fast pulley, and *vice versa*, by a striker attached to the fixed shaft.

The machine has been made to specification, and is supplied by Messrs. White, The Eagle Mills, New Church Road, Camberwell.

South Kensington, October 28, 1902.

# PROCEEDINGS OF SOCIETIES.

## PHYSICAL SOCIETY.

Ordinary Meeting, October 31st, 1902.

Prof. S. P. THOMPSON, President, in the Chair.

A PAPER "On the Existence of a Relationship between the Spectra of some Elements and the Squares of their Atomic Weights," by Dr. W. M. WATTS, was read by Prof. EVERETT.

The author has detected two kinds of relation between the spectra of some allied elements. In the first kind, which is illustrated by comparisons between zinc, cadmium, and mercury, and also between gallium and indium, the differences between the oscillation frequencies of certain lines of one element, are to the differences between the oscillation frequencies of the corresponding lines of another as the squares of their atomic weights. In the second kind the relation is not between two but between three spectra, and is illustrated by the trio potassium, rubidium, and caesium, as well as by the trio calcium, strontium, and barium. The element of greater atomic weight has the smaller frequency; and, in comparing corresponding lines, one from each of the three spectra, the differences of frequency are proportional to the differences between the squares of the atomic weights. If each of the spectral lines in question is represented by a point whose co-ordinates are "frequency" and "square of atomic weight," the three points which represent three corresponding spectral lines will lie on one straight line in the diagram, and these straight lines will be parallel for all the components of a given set of corresponding groups. When a similar mode of plotting by points is employed to exhibit the first kind of relation, the joins of corresponding points meet in a point, which lies on the axis of frequencies; in other words, on the line of zero atomic weight. This relation was indicated by Ramage about a year ago as holding for corresponding doublets and triplets.

Lord KELVIN expressed his interest in the paper.

Prof. PERRY asked to what extent the points actually lay upon straight lines. He had worked with the spectra of potassium, rubidium, and caesium, and knew that many relations which apparently held, on careful examination were found not to be true on account of certain points falling slightly off the curves. He would like to compare the author's results with some of his own work.

A paper on "The Size of Atoms" was read by Mr. H. V. RIBOUT.

This investigation deals with the size of dissociated atoms, or ions, and the results obtained refer to a dissociated atom, as the smallest quantity of matter which can take part in an electrolytic action. The element chosen is hydrogen, and the author concludes that, in round numbers, 1144 million atoms are necessary to form a line 1 c.m. long. The method employed consists in finding a pair of spheres which would be charged by the quantity of electricity known to be necessary to electrolyse a given quantity of the body under examination—in this case water—to the known difference of potential of its ions. From this the size of the atoms is deduced, subject to certain assumptions enumerated and discussed in the paper. The atoms are regarded as spherical and closely packed. To facilitate the calculations, the packing is assumed to be such that the centre of any sphere is immediately above the centre of the sphere upon which it rests. Under these circumstances the total volume of spheres necessary to fill a given cube is equal to that of the single sphere about which the cube is described. The electrical capacities of isolated spheres being proportional to their diameters, it follows that the total capacity of any number of such spheres is equal to the capacity of a single sphere, the diameter of which is equal to the sum of the diameters of the small spheres. Using these

two propositions, the size of the atoms is easily deduced from the pair of spheres already determined. The author points out that the method fixes both the superior and the inferior size of the atoms, and gives, therefore, the true value.

Lord KELVIN remarked that he had often concerned himself with the size of atoms, and pointed out that the value obtained by the author for the diameter of a hydrogen ion was almost exactly one-half of that which he had obtained for the diameter of a molecule of hydrogen. The fact, however, might be a coincidence. He had dealt with a sphere which would have the same effect as a double atom of hydrogen. While avoiding the assumption that atoms are hard and spherical, it was usual to treat them as such for purposes of calculation. The paper was an important one, but there were many assumptions which required looking into. Lord Kelvin said that in dealing with the subject of atoms, it was necessary to consider the atoms of electricity. The atomic theory of electricity, now almost universally accepted, had been thought of by Faraday and Clerk-Maxwell, and definitely proposed by Helmholtz. The atoms of electricity were very much smaller than the atoms of matter, and permeated freely through the spaces occupied by these greater atoms, and also freely through space not occupied by them. An atom of electricity in the interior of an atom of matter experienced electric force towards the centre of the atom. We were forced to conclude that every kind of matter had electricity in it, and Lorenz had named electricity as the moving thing in atomic vibrations. If the electrions, or atoms of electricity, succeeded in getting out of the atoms of matter, they proceeded with the velocity of light, and the body was radio-active. It was therefore not surprising that some bodies showed radio-active properties, but rather surprising that such properties were not shown by all forms of matter. Our knowledge of this subject, which originated with the discovery of the Becquerel rays, had been greatly advanced by the experiments carried out at the Cavendish Laboratory, and he had no doubt that in the next two or three years much light would be thrown upon this important matter.

Prof. EVERETT asked why the author had taken the specific inductive capacity of water equal to two.

The AUTHOR said that the latest determinations of the constant approximated to that number.

Prof. H. L. CALLENDAR exhibited some Vacuum Calorimeters.

Three of the calorimeters were for the determination of the specific heat of mercury, water, and steam respectively by the steady-flow method. The fourth was a vacuum-jacketed Bunsen calorimeter. Prof. Callendar gave some details of the instruments, and described the method of using them.

Miss A. EVERETT exhibited some Photographs of Cross-Sections of Hollow Pencils formed by Oblique Transmission through an Annulus of a Lens.

The direct rays of an arc light were allowed to pass through an annulus of a convex lens tilted to an angle of 45° with their direction, and placed at a distance of about twice its focal length from the arc. The photographic plate was placed at right angles to the beam, and a series of exposures were made at gradually increasing distances from the lens. Two series of photographs were shown, the first series from a plano-convex lens with one annulus, and the second from a double convex lens with two annuli.

The meeting then adjourned until November 14, 1902.

Essence of Cedar-wood from the Atlas Mountains.—Emilien Grimal.—The author prepares an essence from Atlas cedar-wood, an Algerian variety of cedar of Lebanon, by distillation with water. He examines the properties of this substance, and subjects it to a series of fractional distillations. By this means he isolates a cadinene,  $C_{15}H_{24}$ , and a ketone,  $C_{10}H_{14}O$ , which gives the essence its special odour. It also contains traces of ordinary acetone.—*Comptes Rendus*, cxxv., No. 15.

## NOTICES OF BOOKS.

*Chemistry by Observation, Experiment, and Induction.* A Laboratory Manual for Students. By J. I. D. HINDS. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1902. Pp. viii.-192. 12mo. Illustrated.

THE author, who is Professor of Chemistry in the University of Nashville, Tennessee, is already favourably known by his larger treatise, "Inorganic Chemistry," issued by the same publishers. The object of the course outlined in this little volume is "to make the student practically familiar with the properties and reactions of the more important chemical elements and compounds, and to lead him by processes of inductive reasoning to the general principles of theoretical chemistry." The book is of necessity elementary and sketchy; the latter feature is prominent owing to the fact that many of its pages consist of questions about properties and reactions, so printed as to leave blank spaces for the answers to be written in by the students. It seems to the reviewer that the book would have been improved by omitting some of the questions to which the answers are so obvious that they (the questions) are really superfluous. For example, after simple instructions how to prepare, collect, and ignite hydrogen, the question follows:—"Does it burn?" On the other hand, answers to many of the questions cannot possibly be found by the students using this volume, as the questions require knowledge gained elsewhere.

Referring again to hydrogen, three lines below the question already cited occurs this one:—"Heat of the flame?" So far as the reviewer can ascertain by examining the book (there is no index), it contains no information enabling a student to answer this query; the term calories does not occur.

A competent teacher would devise appropriate questions spontaneously, an incapable one would not benefit by a mechanical use of those printed.

The question:—"What does the manganese dioxide do?" when it assists in liberating oxygen from potassium chlorate, might test the knowledge of more proficient students than those for whom the book is intended.

The author uses the spelling adopted by the Chemical Section of the A.A.A.S.; "oxid," "chlorin," "sulfur," &c., a system gradually obtaining more and more favour in the United States.

Used in connection with the larger work of Dr. Hinds this little book may be found helpful.

The volume is well printed and generally free from typographical errors, but "glucenum" (p. 154) has slipped past the eyes of the proof-readers. H.C.B.

*Text-book of Electro-chemistry.* By SVANTE ARRHENIUS. Translated by JOHN MCCRAE, Ph.D. London, New York, and Bombay: Longmans, Green, and Co. 1902.

THIS book is based upon a series of lectures delivered at the University of Stockholm in the autumn of 1897. The German translation, from which this English edition has been prepared, contained considerable additions, the text having been brought up-to-date as far as possible. In the initial chapters, which include a short historical sketch, the fundamental principles underlying the subject are carefully and clearly explained. After considering the general laws relating to the boiling- and freezing-points and vapour pressures of solutions, the author passes to the theory of electrolytic dissociation, and the laws regulating the equilibrium between two phases of a heterogeneous system. The last five chapters are occupied with the calculation of electro-motive forces, potential difference, electro-analysis, and the development of heat by the electric current. The treatment of the subject is most able throughout, and nothing but praise can be accorded to the book, which provides a complete course of electro-chemistry for the student who requires a treatise of moderate difficulty. A useful table of literature references is appended.

*Uebungsbeispiele für die Elektrolytische Darstellung Chemischer Präparate* ("Practical Examples of Electrolytic Methods of Preparation of Chemical Substances"). By Dr. KARL ELBS. Halle-a-S.: Wilhelm Knapp. 1902.

IN the preface to this little book the author points out that up to the present time there has been a great scarcity of books dealing exclusively with electro-chemical methods of preparation of chemical substances. Hence he felt that a collection of suitable examples of such methods would supply a want. The book, which follows the lines of the introduction to electro-chemistry worked through by the students in the University laboratory at Giessen, appears to be admirably suited to its purpose. The student is supposed to be familiar with the use of electrical instruments, and methods of taking simple electrical measurements, this portion of the subject being only very briefly treated in the introduction. Half the book is occupied with methods of preparation of organic compounds, classified under the headings of (1) the electrolysis of organic acids; (2) electro-chemical methods of reduction; (3) electro-chemical methods of oxidation. The experiments are exceedingly well selected, and practical working details are given in all cases. The full explanations with structural formulæ leave no stone unturned to aid the student in acquiring a thorough knowledge of the subject.

*Introductory Chemistry for Intermediate Schools.* By LIONEL M. JONES, B.Sc., A.R.C.Sc. (Lond.). London: Macmillan and Co., Ltd. New York: The Macmillan Co. 1902. Pp. 195.

THE earlier chapters of this book are devoted as much to physical as to chemical work, and are written in an interesting manner so as to hold the attention of the student. In the simple examination of common substances the student is recommended first of all to note all he can detect by the aid of sight and feeling, whether the material is in lumps or powder, glassy and crystalline, or dull and amorphous, &c. Colour, odour, but *not* taste, are also to be noted; such instructions as these will help a boy to observe many things that would otherwise pass unnoticed, and will be of great use in subsequent work. The author has restricted the scope of the book to what may be called the common objects met with, such as chalk, air, water, coal-gas, &c., but has not taken the reader as far as chemical analysis.

At the end of each chapter there is a list of questions to be answered, and numerous practical exercises are given throughout the book.

*Aids to the Analysis and Assay of Ores, Metals, Fuels, &c.* By J. J. MORGAN, F.I.C., F.C.S. London: Baillière, Tindall, and Cox. 1902. Pp. 105.

AS its title indicates, this book is intended for the use of students in practical metallurgy as a laboratory guide. Only the principal well-tried methods of analysis are given, but of these the descriptions will be found to be clear and ample.

Both the wet and dry assay methods are given for all the principal metals and alloys, the latter including silico-spiegel, ferro-tungsten, ferro-aluminium, ferro-nickel, &c., while the analysis of fuels include those in both the solid, liquid, and gaseous forms. The book will be of undoubted help to students.

**Royal Institution.**—The annual course of Christmas Lectures, specially adapted to young people, at the Royal Institution, will be delivered by Professor H. S. Hulse-Shaw, LL.D., F.R.S., Professor of Engineering in University College, Liverpool, whose subject is "Locomotion—on the Earth, through the Water, in the Air." The first lecture will take place on Saturday, December 27, at 3 o'clock, and the remaining lectures will be delivered on December 30, 1902, and on January, 1, 3, 6, and 8, 1903.

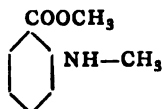
## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxxv., No. 15, October 13, 1902.

**Pentafluoride of Iodine.**—Henri Moissan.—Iodine unites directly with fluorine, the reaction being accompanied by evolution of heat, and the pentavalent compound  $IF_5$  formed. This fluoride possesses great chemical activity. A large number of simple substances decompose it, and in many cases various double reactions take place. When heated to  $500^\circ$  this pentafluoride is decomposed, with evolution of iodine vapour. This phenomenon, however, is only slightly accelerated by an elevation of temperature. The decomposition can be explained either by the fact of a dissociation into fluorine and iodine, or by the presence of free iodine and the production of a new iodine fluoride. The author is continuing his researches on this subject.

**Methyl Methylanthranilate in Vegetable Organisms.**—Eugène Charabot.—The author's researches lead him to conclude that the essence of the leaves of the mandarine orange tree contains about 50 per cent of methyl methylanthranilate—



Up to the present time appreciable quantities of amido-acid ether have never been discovered in the essential oils. The abundance of methyl methylanthranilate found in one organ like the leaf is of great physiological importance and leads to the conclusion that this body ought to play an interesting part in the process of assimilation of mandarine orange tree.

**A New Reaction of Formaline, by which it may be Detected in certain Food-stuffs.**—MM. Manget and Marion.—The use of formaline is becoming more and more general for the preserving of food-stuffs, so the efforts of chemists are directed to finding the most susceptible tests for its presence. The most usual method is to act on the product of distillation. The authors' method has the advantage of acting directly and being more sensitive. It is described shortly as follows:—1. *For Milk.*—Lightly powder the surface of the milk with amidol or amidophenol, and leave this for some moments. Normal milk, or that containing boracic acid, develops a salmon colouration; milk containing formaline, a light yellow colouration. This test is sensitive for 1/50,000th part of formaline. 2. *In Meat Jellies.*—Place a little of the liquefied jelly in a tube, and add some crystals of amidol; shake up. If formaline is present, a yellow colouration is produced, turning to a dirty yellow on the addition of a drop of ammonia. When the jelly contains no formaline, a brownish rose-colour is produced, turning to blue under the same conditions.

## MISCELLANEOUS.

**Royal Institution.**—A General Monthly Meeting of the Members of the Royal Institution was held on the 3rd inst., Sir James Crichton-Brown, Treasurer and Vice-President, in the Chair. The following were elected Members:—Mr. G. H. Baillie, Mr. W. D. Butcher, Mrs. A. R. Cox, Sir Archibald Campbell Lawrie, Mr. G. J. Morrison, and Mr. A. B. Tubini. The special thanks of the Members were returned to Sir Andrew Noble, Bart., K.C.B., F.R.S., for his donation of £150, and to Dr.

Ludwig Mond, F.R.S., for his donation of £200 to the Fund for the Promotion of Experimental Research at Low Temperatures.

**Institute of Chemistry of Great Britain and Ireland.**—The next Examinations of the Institute of Chemistry, for persons desirous of qualifying themselves to be Public and Technical Analysts, will be held at 30, Bloomsbury Square, London, W.C., in January, 1903. The Examinations are open only to candidates who have complied with the Regulations. The Intermediate Examination will occupy at least four days, commencing on Tuesday, January 6th. Examinations in the following Branches of the Final Examination for the Associateship (A.I.C.) will extend over at least four days, and may commence on either January 6th or 13th:—(a) Mineral Chemistry, (b) Metallurgical Chemistry, (c) Physical Chemistry, (d) Organic Chemistry, and (e) The Analysis of Food and Drugs, and of Water, including an Examination in Therapeutics, Pharmacology, and Microscopy. (The Final Examination in Branch (f), Biological Chemistry, is held in October each year). Applications for admission to the January Examinations should be forwarded to the Registrar not later than Tuesday, December 2nd, 1902, on which day the List of Candidates will be closed.

**Apparatus and Method for Exact Incineration.**—H. Wislicenus.—The most satisfactory method for the estimation of the ash of an organic body is that of Wackenroder, viz., incineration in the presence of acetate of lime. This method has the advantage of allowing the complete solution of the ash in hydrochloric acid, and prevents the expulsion of the volatile metalloids by means of the silica or carbon. It is prudent, nevertheless, to add milk of lime to the acetate; this will maintain the excess of alkalinity. The carbonisation is effected easily, but the total combustion of the carbon necessitates the use of certain adjuvants, of which peroxide of hydrogen at 3 per cent is most to be recommended. The material is moistened, dried on the water-bath, and then calcined. The error due to the carrying off of solid particles by the gases and the steam can only be prevented by the help of a special apparatus. That proposed by the author consists of a platinum cover which can be fitted to an ordinary crucible, carrying an escape-tube through which air is drawn. The solid particles carried off by the air are arrested by passing through milk of lime, which is added to the contents of the crucible after the operation. The author recalls another method of calcination which is not very well known, and was devised by Grouven, and which consists in operating in a current of steam between  $400^\circ$  and  $700^\circ$ . The ashes obtained are very pure; they differ from ashes burnt in air, inasmuch as the organic sulphur has disappeared by volatilisation, which is a great advantage in certain cases.—*Zeit. Anal. Ch.*, vol. xl., p. 441.

**The Ammoniacal Compounds of Sulphocyanide and of Isosulphocyanide of Copper.**—M. Litterscheid.—Sulphocyanide of ammonia and copper,  $\text{Cu} \begin{array}{c} \text{NH}_2-\text{CNS} \\ | \\ \text{NH}_2-\text{CNS} \end{array}$  is obtained in the crystalline form, when dilute solutions of ammonia and sulphocyanide of ammonia are brought into contact with a solution of sulphate of copper. The purified salt gives an analysis corresponding with the above formula. A compound richer in ammonia is obtained when we treat a concentrated solution of sulphate of copper with ammonia at 25 to 30 per cent, in the presence of a sufficient quantity of  $\text{NH}_4\text{CNS}$ . Analysis gives the following formula:— $\text{Cu} \begin{array}{c} \text{NH}_2-\text{CNS} \\ | \\ \text{NH}_2-\text{CNS} + 2\text{NH}_3 \end{array}$ . The author has not succeeded in isolating a compound analogous to the isosulphocyanide. It is certainly formed when the isosulphocyanide is heated for a prolonged period with ammonia. In fact, it separates out in beautiful brilliant needles, which cannot be isolated, however, without becoming decomposed rapidly; this fact renders its analysis impossible.—*Arch. d. Pharm.*, vol. ccxxxix., p. 336.

## NOTES AND QUERIES.

\*. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

**Sodium Bismuthate.**—(Reply to S. C.)—The preparation is described in the original paper of Reddrop and Ramage, which appeared in the *Transactions of the Chemical Society*, April, 1895, p. 271.—H. GRIPPER, G.C.R., Gorton, Manchester.

**Sodium Bismuthate.**—S. C. will find the necessary particulars quoted in Brearley and Ibbotson's "Analysis of Steel-works Materials" (Longmans, Green, & Co.)—J. T.

## MEETINGS FOR THE WEEK

FRIDAY, 14th.—Physical, 5. "Theory of the Aluminium Electrode," by Dr. W. W. Taylor and J. K. H. Inglis. "Determination of the Ratio of the Specific Heats at Constant Pressure and at Constant Volume for Air and Steam," by W. Makower.

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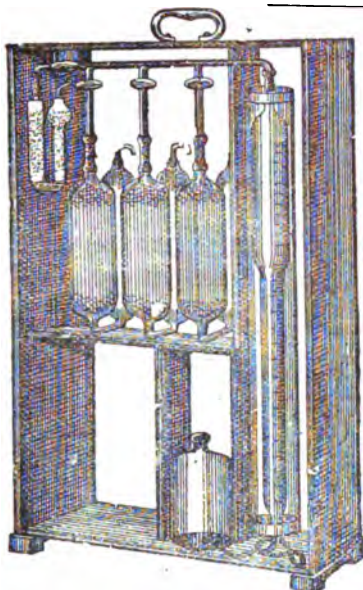
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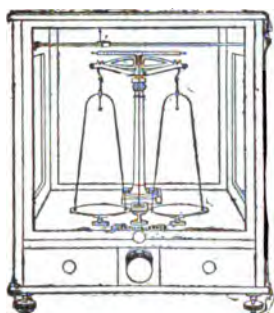
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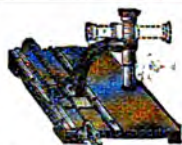
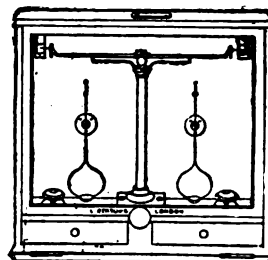


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THE CHEMICAL NEWS.

VOL. LXXXVI., No. 2242.

SOME INTERESTING PROBLEMS RELATING TO DAIRY WATER SUPPLIES.

By J. BRISTOWE P. HARRISON, F.I.C.

NOT the least important of the many precautions which are exercised in the control of a large dairy, having the slightest claim to being conducted on the most modern scientific principles, is the regular inspection of the water supplying its many farms, in order to render the risk of contaminating the milk at this stage of its existence as small as possible. In the pursuance of such a system, it is not surprising that some rather interesting—not to say difficult—problems should occasionally present themselves; and in the course of my duties as Assistant Chemist to the Aylesbury Dairy Co., Ltd., it has occurred to me, on more than one occasion, that some of the more difficult problems relating to the supplies of that Company's various farms might be of sufficient interest to dairy chemists, public analysts, and others interested in water analysis to justify their publication.*

For the purposes of this paper, I shall denote the examples mentioned as A, B, C, &c. The time of analysis is placed at the head of each set of results, which are expressed in parts per 100,000.

A common idea which prevails with the owner of a water supply is that once a water is analysed and found to be good, its composition will remain the same to the end of all time. It is only with very great difficulty that you can convince him of the necessity for regular examination, in order to make sure that his supply is proof against contaminating influences.

The results obtained from the analyses of the supplies A, B, and C show how such a water may change in character from time to time, and how imperative it is that the report of the expert should be guardedly expressed when giving his opinion as to the quality of a supply for the first time.

"A" is a well about 60 feet deep to the surface of the water, and apparently far removed from possible sources of pollution. From the results of the first analysis (June, 1896) the water was considered satisfactory, but on re-examination there was distinct evidence of surface pollution. After due precautions had been taken to prevent surface leakage, the water appeared to be changing in character, and its use had to be finally prohibited. It will be noticed that the chlorine figure is high during the year 1899, which was remarkable for its dry summer, and that, while the chlorine increases slightly, in other respects the composition of the water improves. After a very careful consideration of the results obtained at different times, it was concluded that the well was probably supplied by water from at least two different strata—one low in chlorine, the other very high. The low chlorine water is probably the upper one, and in seasons of drought may become exhausted, when the well is fed by the lower supply, mixed with polluting matter which has percolated through the cracked stratum.

The supply "B" is a well about 30 feet deep, and was at first reported as far removed from possible sources of pollution. As the water was constantly varying its composition, a further inspection of the surroundings was made, and revealed the presence of a cesspool. The well was opened, and it was found that the sides were quite

pervious, and therefore liable at any time to let in surface pollution.

"C" was a new well 50 feet deep, and sunk through a layer of rock. To all appearances it was admirably placed, and water was pumped out until the composition of the supply was practically constant, as indicated by the analysis headed November, 1900. The water, for the time being, although rather high in sulphates, was considered satisfactory. Unfortunately, on standing, however, its composition was again found to be unfavourable, owing, no doubt, to the leakage through of surface water, which had accumulated above the solid rock in the loose sub-soil.

Notwithstanding the rapid progress that has been made of late years in other departments of engineering, the well-sinker in the country has still his own ideas as to how a well should be constructed. It is apparently a matter of indifference to him how deep it should be, and at what part of the well the water should enter. He also seems to have a kind of impression that the nearer the well is to some inhabited dwelling, the more convenient it will be for those who use it. The result of all this is that wells are generally sunk in close proximity to inhabited houses, with their defective drains, privies, and cesspools, and impurities from these sooner or later find their way into the water supply. No wonder, then, that so many country wells contain impure water, which are obviously liable at any time to be a source of danger to the public health.

A perusal of the analyses of the supplies D, E, and F will at once make plain that an ideal well should be sunk as deeply as possible and at some considerable distance from any source of pollution, and should also be protected from surface leakage by an impervious stratum. D and E are proximate wells on opposite sides of a road, and undoubtedly fed by the same water, whereas F is a supply altogether distinct from these.

The tube-well "D" is 30 feet deep and reasonably removed from anything which may contaminate it; it penetrates 3 feet into the chalk. From August, 1898, to April, 1902, the water from this well was analysed no fewer than ten times, and its analysis remained almost constant during the whole of that period.

"E" is about 20 feet deep, and situated at the upper corner of a farm-yard at a somewhat lower level than "D." Since it was first analysed it has been constantly changing its composition, but this change has been so small and has taken place so slowly that, so far as the chemical analysis was concerned, the first indications of pollution might have been entirely overlooked if the composition of the supply "D" had not been available as a standard to judge from.

The nitric acid of these two supplies is undoubtedly derived from the chalk. We have here an excellent illustration in which a bacteriological examination is of more value in indicating pollution than a chemical one. The chief point to notice, however, is that one of these supplies being a tube-well and dipping well into the chalk, is rendered impervious to contamination, while the other, standing in the corner of the farm-yard, is always liable to slight leakage.

The supply "F" was thus described:—"The water emerges from a sand spring not far from the top of a hill. It is collected and conveyed by iron pipes through a wood down the side of the hill to a closed reservoir at the bottom. There appears to be no possibility of pollution once it reaches the reservoir."

Such conditions would usually be considered ideal for a water supply, and seeing that it had previously been analysed and reported as pure, it was somewhat of a puzzle to find that on each occasion when the water was examined, the analysis invariably turned out unfavourable. An inspection of the locality by the chemist, however, revealed that a village was situated on the brow of the hill, and that the sand-spring was fed by a number of small brooks which skirted the village. Remembering that the water from the spring was practically surface water, it

* As the features of a good water are of little interest from a scientific point of view, it is hardly necessary to point out that I have purposely chosen for example supplies which were polluted, and the use of which was, of course, prohibited.

	A.					
	June, 1896.	May, 1899.	June, 1899.	August, 1899.	March, 1900.	May, 1901.
Chlorine	1'25	22'10	22'90	23'50	22'9	1'45
Free ammonia	0'003	0'0045	0'005	0'008	0'0095	0'004
Albumenoid ammonia	0'005	0'047	0'0145	0'010	0'017	0'0085
Nitrates	1'0	3'8	1'9	None	2'5	0'8
Nitrites	None	None	None	None	Trace	None
Oxygen absorbed in four hours	0'022	0'118	0'135	0'140	0'120	0'019
Phosphates	Trace	Trace	Trace	Trace	Trace	Trace

Bacteriological Examination.

Gelatin culture at 22°, colonies per c.c.	—	3640	—	1310	50	—
Agar culture at 37°, colonies per c.c...	—	Plate covered in 48 hours.	—	3	4	—

	B.				
	September, 1897.	November, 1898.	April, 1899.	July, 1899.	October, 1899.
Chlorine	4'1	4'7	4'8	6'0	4'75
Free ammonia	0'001	0'004	Trace	None	None
Albumenoid ammonia	0'006	0'033	0'0195	0'009	0'011
N ₂ O ₅	None	1'2	2'2	1'1	0'2
N ₂ O ₃	None	None	None	None	None
Oxygen absorbed in four hours	0'032	0'057	0'039	0'046	0'042
P ₂ O ₅	None	None	Trace	Trace	Trace

Bacteriological Examination.

Gelatin culture at 22°, colonies per c.c.	—	620	1260	—	Plate covered.
Agar culture at 37°, colonies per c.c...	—	88	42	—	240

	C.				
	April, 1900.	May, 1900.	June, 1900.	November, 1900.	April, 1901.
Chlorine	1'9	2'4	1'9	1'9	2'35
Free ammonia	0'0065	0'008	None	0'0045	0'0045
Albumenoid ammonia	0'0095	0'013	0'009	0'007	0'0115
N ₂ O ₅	None	Trace	None	None	0'75
N ₂ O ₃	None	None	None	None	None
Oxygen absorbed in four hours	0'027	0'009	0'131	0'021	0'065
SO ₂	32'4	62'4	53'0	49'4	42'0
P ₂ O ₅	Trace	—	—	—	—

Bacteriological Examination.

Gelatin culture at 22°, colonies per c.c.	—	950	—	240	12,360
Agar culture at 37°, colonies per c.c...	—	5	—	None	4

	D.					
	August, 1893.	Sept., 1897.	Nov., 1898.	Nov., 1899.	Nov., 1901.	May, 1902.
Chlorine	1'35	1'35	1'3	1'35	1'35	1'30
Free ammonia	0'002	None	0'0045	None	None	None
Albumenoid ammonia	0'005	0'002	0'004	0'004	0'005	0'0045
N ₂ O ₅	2'0	2'1	2'8	1'8	2'0	1'8
N ₂ O ₃	None	None	None	None	None	None
Oxygen absorbed in four hours	0'018	0'007	None	0'007	0'003	0'015
P ₂ O ₅	None	None	None	None	—	—

Bacteriological Examination.

Gelatin culture at 22°, colonies per c.c.	—	—	None	350	10	530
Agar culture at 37°, colonies per c.c...	—	—	None	1	1	None

	E.					
	August, 1893.	Sept., 1897.	Nov., 1898.	Nov., 1899.	Nov., 1901.	May, 1902.
Chlorine	1'35	1'5	1'55	1'5	1'55	1'5
Free ammonia	0'003	0'005	0'003	Trace	0'003	None
Albumenoid ammonia	0'005	0'005	0'0045	0'0055	0'005	0'0065
N ₂ O ₅	2'0	2'1	2'8	2'3	2'0	1'9
N ₂ O ₃	None	None	None	None	None	None
Oxygen absorbed in four hours	0'002	0'027	0'0335	0'028	0'021	0'032
P ₂ O ₅	None	Trace	Trace	Trace	—	—

Bacteriological Examination.

Gelatin culture at 22°, colonies per c.c.	—	—	280	7480	1670	Large
Agar culture at 37°, colonies per c.c...	—	—	Plate covered in two days by <i>B. ramosus</i> .			

F.

Chlorine	3'45
Free ammonia	0'0031
Albumenoid ammonia	0'0130
N ₂ O ₅	7'1
N ₂ O ₃	None
Oxygen absorbed in four hours	0'05
P ₂ O ₅	Trace

Bacteriological Examination.

Gelatin culture at 22°, colonies per c.c.	120
Agar culture at 37°, colonies per c.c.	Whole plate covered in less than 24 hours.

G.

	November, 1896.	November, 1897.	May, 1899.	March, 1900.	November, 1901.
Chlorine	0'95	1'20	1'20	1'20	1'20
Free ammonia	0'002	0'003	0'0025	None	0'002
Albumenoid ammonia	0'010	0'014	0'0145	0'010	0'008
N ₂ O ₅	1'5	1'5	1'2	1'5	0'85
N ₂ O ₃	None	None	None	None	Trace
Oxygen absorbed in four hours	0'016	0'053	0'028	0'013	0'030
P ₂ O ₅	Trace	Trace	None	—	—

Bacteriological Examination.

Gelatin culture at 22°, colonies per c.c. —	6620	3340	2000	840
Agar culture at 37°, colonies per c.c. —	None	<i>B. ramosus</i>	<i>B. ramosus</i>	43
Phenol agar culture at 37°, colonies per c.c. —	None	—	—	—

H.

Free ammonia	0'040	Magnesium sulphate	48'00
Albumenoid ammonia	0'010	Sodium sulphate	268'19
Oxygen absorbed in four hours	0'170	Sodium chloride	49'96
Calcium nitrate	3'04	Potassium chloride	30'85
Calcium carbonate	52'41	Silica	2'10
Calcium sulphate	35'41		

was not very difficult to understand where the pollution was coming from.

This water was analysed four times, and though the analyses varied slightly at each examination, the general character of the water showed no appreciable change. There is evidently no object in recording each separate analysis, so I give the mean of four analyses (see F).

A village with no satisfactory water supply in its own immediate neighbourhood, notwithstanding the many attempts to obtain one by sinking wells, is compelled to cart water from a well on a hill 2½ miles away to obtain a pure water supply. The locality of the well has been thoroughly inspected; the gathering ground is entirely uninhabited, and there is no reason to believe that the supply is other than a good one. The analyses of "G" are a series of examinations of this water which has been carted away in an open tank. For reasons which need not here be considered, it was necessary to analyse this water as received at the village, and not as taken directly at the source. In quoting the following analyses I would specially draw attention to the unfavourable bacteriological indications, which are undoubtedly due to what the water received on its journey in the open tank, and not to impurities in the water as taken from the well. Under such circumstances, it is hardly necessary to point out how misleading such indications would be if considered from an absolute standpoint.

The following case is interesting, yet hardly credible in these days of stern fact. It serves to show, however, what a remarkable influence the water finder and his magic rod still have upon the unsuspecting British farmer:—A certain farmer, after spending a considerable sum of money in vain attempts to find a satisfactory water supply, succumbed to the assurances of a water finder who possessed a considerable reputation. They accordingly set out on their mission, and had not gone very far before the farmer reminded his companion that they had just passed over water. "Yes," replied the finder, "but it is not good water; what I undertake to discover is a good potable water; the divining-rod will not indicate the presence of bad water." The farmer having remarked that the water they had just passed over was "brackish,"

they pursued their course until the hazel-twig "divined" what the finder considered a good potable water. I give the analysis of this (see H); its composition agrees so closely with that of the "brackish" water that there can be no question about their being one and the same. *Verbum sap.*

THE ESTIMATION OF ALCOHOL IN VERY DILUTE SOLUTIONS.

By G. ARGENSON.

THE method I am about to describe enables us to estimate ethylic alcohol in solutions containing only 1 part in 1,000,000 by volume. The principle of the method is as follows:—The alcoholic solution (very dilute of course), of which we wish to know the strength, is treated with chromic mixture and distilled; under suitable conditions the greater part of the alcohol is oxidised to the form of aldehyde, which is found in the first portions which come over on distillation.

If we add a few drops of an aqueous solution of fuchsin, decolourised by sulphurous acid, to the distillate, a violet colouration is obtained, the intensity of which depends on the amount of aldehyde contained in the liquid, and, consequently, on the amount of alcohol in the original solution. As this colouration is very similar to that of dilute solutions of permanganate of potassium, its intensity is estimated in the following manner:—

To a known volume of water, a titrated solution of permanganate is added, drop by drop, until the colouration obtained is of the same intensity as that of the solution under examination; from the volume of the solution of permanganate used we can calculate the amount of alcohol present, by means of preliminary trials on solutions of known strength.

The sensitiveness of this method depends on that of the reagent used; to obtain the greatest delicacy it is best to proceed in the following manner:—Twenty-five grms. of fuchsin are dissolved in a little less than 500 c.c. of water, from which all the dissolved air has been driven off

by boiling; the solution takes place more rapidly in hot than in cold water; after cooling, the volume is made up to 500 c.c., and a very slow current of sulphurous anhydride is passed through; the current of gas is stopped before the decolouration is complete, as the action still goes on with the lapse of time. After a few hours, if the solution is still coloured, a few more bubbles of gas are passed through, and we wait again for a few hours longer, and so on, taking care to stop while the solution still has a faint rose tint. If the operation was pushed further, we should obtain a straw-coloured solution, the sensitiveness of which would be considerably less. When prepared in this manner and used as I am about to show, this reagent will give an appreciable colouration with a solution containing 1 part of alcohol per 10,000,000 by volume.

This reagent will remain intact for several months, if kept in full bottles away from the light.

The reagent made, we do a preliminary determination by operating on a solution of alcohol of known strength, 1 in 500,000 by volume, for example.

For this purpose, we take 20 c.c. of this solution and place it in a flask; 5 c.c. of a saturated solution of bichromate of potassium, and 1 c.c. of concentrated boiled sulphuric acid are added. The flask is placed in communication with a small Liebig's condenser by means of an elbow tube, of which the vertical portion has two or three bulbs blown in it to arrest any projections during distillation; this must be conducted slowly, and always in the same manner. The first 5 c.c. are collected in a test-tube A, marked at this point, and 0.5 c.c. of the reagent is added. The colouration appears gradually, and after about an hour it may be considered permanent. This is the moment for making the comparison of the tint, which is carried out as follows:—Five c.c. of water are placed in a test-tube of the same diameter as the graduated one; then, by means of a small burette, we add a centinormal solution of permanganate, first drop by drop, and then by fractions of a drop, until the colouration is of the same intensity as that in the tube A. The two tubes are compared by transmitted light in front of a sheet of ground glass or oiled paper, and the volume of the solution of permanganate is noted.

The same series of operations is repeated on the liquid in which we wish to know the proportion of alcohol; this latter is practically proportional to the volume of the solution of permanganate necessary to obtain equal tints, for proportions of alcohol comprised between 1 part in 200,000 and 1 part in 100,000 under the conditions described. In many cases, knowing how extremely dilute the solutions under examination are, we may be content with the approximation obtained by applying the rule of proportions, although the relative error may be as great as one-third. If we wish for a closer approximation, the experiment can be repeated with a solution of a strength more closely resembling it than the one first used, and the new figure expressing the volume of the permanganate will serve to correct the first; the relative proportion of this to the volume of alcohol present may be looked upon as correct for solutions of about the same composition.

In the case of solutions containing more than 1 part of alcohol in 200,000 by volume, the reagent will give a colouration of an intensity equal to, or greater than, that of the centinormal solution of permanganate. In such a case the experiment must be repeated after having diluted the solution to a known extent, to bring it within convenient limits.

The centinormal solution of permanganate should be prepared at the time it is required for use; it is advisable to use a burette of very small diameter reading to $\frac{1}{100}$ th of a c.c.

We may have occasion to estimate the alcohol in a solution which also contains aldehyde; this must be verified by means of the reagent before treating it with the chromic mixture; this aldehyde, added to that formed by the oxidation of the alcohol, would give too high a figure.

The cause of error must be allowed for by distilling 20 c.c. of the solution without treating it with the chromic mixture and continuing the operation as described above. The volume of the permanganate solution must be deducted from that found by the first operation, and the difference will give the figure corresponding to the alcohol only.

We may remark here that alcohol generally contains traces of aldehyde which sometimes give a very intense colouration. I have tried, but without success, to eliminate these traces by acting on the alcohol for a long time with ammoniacal nitrate of silver, potash, and baryta. I have also tried to obtain alcohol free from aldehyde by saponifying, by means of dilute sulphuric acid, the sulphovinate of soda dried by keeping in the oven for several days, taking care to do the saponification in an apparatus from which all the air has been driven by means of a current of carbonic acid.

As a general rule, the amount of aldehyde is sufficiently small for the very dilute solutions used for titrating not to give any appreciable colouration with the most sensitive reagent; this is quite pure enough for ordinary practice.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 18.

"FIXING" NITROGEN FROM THE ATMOSPHERE.

ONE of the first results of Sir William Crookes's Presidential Address to the British Association, in the year 1898, was a feeling of scepticism, followed by alarm, as to the serious extent to which the world's wheat supply is threatened by the failing fertility of the available soil. The principal object of that Address was overlooked by most of its critics, viz., to point out how the threatened failure of the wheat supply could be averted. It was not recognised at that time that this feeling of alarm for the future was emphasised in order to lead up to the remedy which Sir William Crookes had to propose, viz., to augment the supply of nitrates for manurial purposes by the fixation of the atmospheric nitrogen by means of electricity. Fortunately, however, the matter was not lost sight of, and we read now in a paper on this subject by Mr. T. C. Martin, in the September number of *The American Monthly Review of Reviews*, that the manufacture of nitrates from the nitrogen of the atmosphere is in a fair way to become a commercial fact.

It was calculated that in about thirty years' time some 12,000,000 tons of nitrate of soda would be required annually to raise the necessary supply of wheat. The nitrate deposits in Chili are swiftly running out, and the amount now in sight and available is estimated to be good for only fifty years at the present rate of consumption. Hence, even if famine does not impend immediately, the food problem is far more serious than is generally supposed.

Dealing with the conditions as they are, Sir William Crookes pointed to an inexhaustible supply of nitrogen which has now been tapped by the method we are about to describe.

This new industrial enterprise has been founded at Niagara Falls by Messrs. Charles S. Bradley and D. R. Lovejoy, and the method which they have devised consists mainly in the production of a large number of electric arcs or flames in a confined space, through which a regulated amount of air to be burned could be passed continuously; this air emerging from the apparatus laden with nitric oxides, as the result of combustion, is ready for treatment and collection.

A great deal of time and money had to be spent in determining the form or variety of arc that would effect the maximum chemical union of the nitrogen and oxygen in the air. The curious discovery was made at an early stage that "static" sparks, such as are caused by frictional machines, are of very little use for this work; in

fact, there was not enough energy in them. Turning, therefore, to dynamic electricity, the two inventors ascertained that for their work, an alternating current arc was not so good as the direct current arc, and a dynamo was specially wound, giving a direct current at a pressure of 10,000 volts, the power being obtained from Niagara.

The nitrifying chamber in which the arcs are situated consists essentially of a large box of metal, six feet high and three feet in diameter; inside, there is a revolving cylinder or hollow shaft. The box has openings to admit and circulate the air, and all round the walls are rows of fixed electrical contacts for arcing points, arranged in six rows of twenty-three each, or 138 in all. The positive pole of the dynamo is connected in "multiple" or "parallel" to these points, with an induction coil in each circuit so as to prevent the arcs from "short-circuiting" and burning out the dynamo.

The negative pole of the dynamo is connected with the revolving member inside the chamber; this cylinder having contact projections, or wire fingers, corresponding to the contacts on the inside of the box, so that as it spins round, the currents from the 10,000 volt dynamo cross in the air between the stationary contact points and the revolving ones. The arcs are thus rapidly made, broken, and re-made within the chamber. A motor at the top of the apparatus drives the cylinder at a speed of from 300 to 500 revolutions per minute, while air is forced into the chamber at the rate of 5 cubic feet per arc contact per hour. The air leaves the chamber after this treatment laden with $\frac{2}{3}$ per cent of oxides of nitrogen. The points of contact on the barrel are set each a little in advance of the next so as to form spirals from the top to the bottom of the shaft, enabling the arcs to work on the air without cessation. The arcs themselves are six or eight inches long, which is very much longer than the arc usually used for lighting purposes. From this chamber the acted on air is led away to an absorption tower of the kind generally used in chemical processes, the result of this treatment being nitric and nitrous acids. If the gases are brought into contact with potash, saltpetre is formed; if the base is caustic soda, nitrate of soda is formed.

A careful investigation of this process has shown that, with current at Niagara costing £4 per kilowatt—i.e., 1½ horse-power—per year, the expense for the energy required would be a little less than 2d. per pound of 70 per cent nitric acid, which is the customary commercial strength of acid selling at 2½d. per pound. There is every reason to believe that this process will be the means of placing a large supply of nitrate of soda on the market, and thus show that science, while continually increasing our wants by facilitating intercommunication, is also able to help humanity out of a great difficulty.

ALLOYS OF CADMIUM AND MAGNESIUM.

By O. BOUDOUARD.

IN two previous papers (*Bull. Soc. Chim.*, Series 3, vol. xxvii., pp. 5 and 45) I described the alloys of aluminium and magnesium from the point of view of their fusibility, and I also described the definite compounds formed by these metals. I have followed up these researches relating to the alloys of magnesium by the examination of those formed with cadmium.*

Fusibility.—Knowing the low melting-point of cadmium, I was able to use, for the purpose of studying the fusibility of the alloys of cadmium and magnesium, a similar process to that which I described in connection with the

alloys of aluminium and magnesium. The following results were obtained:—

Cd per cent by weight.	Mg per cent by weight.	Temperature.
100	—	320°
90	10	410
85	15	500
80	20	440
75	25	400
70	30	480
60	40	525
55	45	565
50	50	560
40	60	575
30	70	590
20	80	635
10	90	650
—	100	635

If we construct a curve, taking the proportions by weight of magnesium as abscissæ, and the temperatures as ordinates, we observe that this curve has three maxima (500, 565, and 650°) and two minima (400 and 560°). The last maximum shows that the corresponding compound is less fusible than either of its two constituents.

Similar facts have already been observed by Sir Wm. Roberts-Austen (gold-aluminium), H. Gautier (antimony-aluminium), and Lebeau (antimony-lithium). These three maxima are evidence of the existence of three definite compounds, CdMg, CdMg₂, and CdMg₃.

The alloys of cadmium and magnesium are of a more or less brilliant white appearance; they are easily filed, and are fairly soft; when it is wished to prepare the surface for microscopical examination it is difficult to obtain a perfect polish. From the point of view of malleability the alloys of cadmium and magnesium break when they are submitted to continuous hammering; those alloys containing equal parts of cadmium and magnesium behave the best. We may here remark that this property is the reverse of that observed with alloys of aluminium, and with those of copper which we are now investigating; in the latter case especially the fragility of the metals obtained is very great.

Alloys of cadmium and magnesium preserved in stoppered flasks do not undergo any sensible change in the air. The same is not the case in the presence of water; ingots containing 90 parts of cadmium and 10 of magnesium, 50 cadmium, 50 magnesium, and 10 cadmium, 90 magnesium, are attacked during polishing with a moist cloth impregnated with oxide of iron.

Microscopic Metallography.—The preparation of polished surfaces of the different alloys examined offers difficulties due to their want of hardness; it is difficult, not to say impossible, to obtain a perfect polish. Sometimes besides the softness, we are also troubled with the facility with which the alloys are attacked by water.

The methods of etching are extremely various. In a general way, the mineral acids, even when very dilute (up to 1 per cent), act very energetically in a very short time, and to the attack proper is added a superficial oxidation which makes the photographic reproduction very difficult.

However, by partially re-polishing the polished surfaces thus etched we can obtain satisfactory results. Hydrochlorate of ammonium gave me some good results with alloys of magnesium and aluminium; the same was not the case with those of cadmium and magnesium. Etching by means of the electric current was tried with success.

By varying the proportions of magnesium from 0 to 100, I prepared a series of nine specimens which were examined with a Le Chatelier microscope.

Nitric acid at 5 per cent showed the existence of magnificent dendrites of several millimetres in length in the specimen of 90 cadmium, 10 magnesium; they were even visible to the naked eye (compound CdMg); to obtain a photograph it was only necessary to partially polish the specimen thus etched. By simple polishing in bas-relief

* Parkinson, in his work on the alloys of magnesium, shows that on melting cadmium with 10 per cent of magnesium in a current of hydrogen, he obtained a brilliant silver-white metal as hard and as brittle as magnesium, and coarsely crystalline. This metal tarnishes rapidly; after being kept for three months in a sealed tube, it becomes disintegrated (*Chem. Soc.*, Series 2, vol. v., p. 117).

on a moist cloth, the ingot 50 cadmium, 50 magnesium, shows a very distinct crystallisation (compound CdMg_4). Finally, if after polishing the sample 10 cadmium, 90 magnesium, we wash it in water, we obtain very irregular polygonal contours, which become more distinct by treating with 10 per cent chloride of sodium, under the influence of the electric current (compound CdMg_{30}).

Definite Compounds.—I found it difficult to isolate the definite compounds of cadmium and magnesium. The mineral acids used varied in concentration from 20 per cent to 1 per cent; the acetic acid was at 10 per cent, and the hydrochlorate of ammonium at 10 per cent, but none of these gave me any satisfactory results with the compounds CdMg and CdMg_4 .

1st. CdMg .—The centesimal composition of this alloy corresponds to 82.1 of cadmium and 17.9 of magnesium. If the metallic ingot of 25Mg—75Cd is attacked by 1 per cent hydrochlorate of ammonium in the cold, gas is given off, and, at the same time, a crystalline powder of a grey metallic appearance separates; this is collected, and gives on analysis: Cd = 81.2, 83.3; Mg = 18.7, 17.0.

2nd. CdMg_4 .—The centesimal composition of this alloy corresponds to 53.5 of cadmium and 46.5 of magnesium. The best method for isolating the compound CdMg_4 consists in treating the metallic ingot of 50Cd—50Mg by means of 1 per cent hydrochlorate of ammonium. The attack can be carried out either hot or cold, and a crystalline powder is obtained. Analysis gives the following results: Cd = 54.8, 51.0, 53.5, 52.65; Mg = 45.3, 48.8, 46.4, and 47.25.

3rd. CdMg_{30} .—The theoretical centesimal composition of this alloy corresponds to 13.2 cadmium and 86.8 magnesium.

All efforts to isolate the compound CdMg_{30} have been unsuccessful; the results given by analysis have varied considerably according to the method of attack adopted.

I. Attack of the ingot 90Mg—10Cd by AmCl at 1 per cent	37.8, 62.1
II. Attack of the ingot 90Mg—10Cd by AmCl at 1 per cent	26.4, 73.1
III. Attack of the ingot 90Mg—10Cd by AmCl at 10 per cent	98.0, 2.0
IV. Attack of the ingot 70Mg—30Cd by HCl at 5 per cent	64.7, 35.3
V. Attack of the ingot 70Mg—30Cd by HCl at 5 per cent	59.6, 40.3

The apparition of the maximum point in the curve of fusibility may be explained by admitting the existence of solid solutions, either between the cadmium and magnesium, or between the definite compound CdMg_4 and magnesium.

The analyses I. and II. verify this latter hypothesis; they correspond to the compound CdMg_4 in an excess of magnesium. As for the analyses III., IV., and V., they indicate that through the too great energy of the reagents used, a large quantity of magnesium has gone into solution. Finally, the micrographic examination of the ingot 90Mg—10Cd has put in evidence a series of irregular polygonal grains which should be thus constituted, not by an individual chemical compound, but by a solid solution. Facts similar to these have been observed with alloys of copper and antimony; the results obtained by M. Le Chatelier in the examination of the fusibility and dilatation of these alloys, those of Mr. Stead from the micrographic point of view, and those of M. Kamensky relative to the electrical resistance, establish in fact, the existence of a solid solution in the alloys of copper and antimony.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 16.

Pulegenic Acid.—L. Bouveault and L. Tetry.—The authors have repeated M. Wallach's experiments of obtaining pulegenic acid by the reaction of alcoholate of soda on bibromide of pulegon, but they found that, besides this acid, other interesting products are formed.—*Bull. Soc. Chim.*, Series 3, xxvii., No. 8.

ON THE

RAMIE (*BOEHMERIA*) PLANT AND CROP.

By R. W. EMERSON MACIVOR, F.I.C., &c.,
Formerly Assistant Professor of Chemistry, Anderson's College
(Medical Faculty), Glasgow; Consulting Chemist to the National
(now Royal) Agricultural Society, Victoria, Australia, &c.

RAMIE is the bast fibre of several varieties of the genus *Boehmeria* (*Urticaceae*), but ordinarily of the *B. nivea*. The demand for this valuable material is rapidly increasing year by year, particularly in France and Germany, and must sooner or later cause the cultivation of the crop to extend in every country where the climate, soil, labour conditions, and other circumstances are at all favourable to the industry. It is much to be regretted that we are so far behind our Continental friends in regard to this important textile. The difficulty our manufacturers have hitherto had in obtaining constant and abundant supplies of the raw material has, I am informed, prevented the general adoption of ramie spinning in this country. No doubt when this fact becomes more generally realised by our colonial compatriots in the languishing West Indian Islands, Fiji, and other tropical possessions, and substantial prospects are held out to them in the matter of steady markets for their produce, we may hope that they will enter upon the general cultivation of the crop, with incalculable advantage to their respective countries and the whole Empire.

The progress of ramie growing was for many years delayed through the lack of proper means of stripping the fibre-containing bark from the underlying useless wood of the stalk. This difficulty has, however, been overcome, and the bark is sent into the market in the form of "ribbons," or "brown" ramie or rhea. In order to free the fibre from the so-called "gum," pectic, and other substances with which it is naturally associated, the ribbons are subjected to the operation known as "de-gumming." The processes which have been proposed to effect this end are very numerous, and the greater number of them involve the exposure of the ribbons to heated alkaline solutions (usually caustic soda) and acid baths of one description and another for periods of time varying much, according to the nature and strength of the solutions employed and the condition of the material under treatment. It is very generally believed that the chemicals ordinarily employed—caustic soda particularly—in "de-gumming" have the effect of breaking up the fibre in such a manner that it becomes short and "yields a weak product in commercial usage." In some instances, in which the solutions used were, through ignorance on the part of the operators, too strong, I have found this to be only too true; but, unfortunately, the breaking up also occurs to some extent in processes where neither acids nor alkalis are employed. Possibly, as the long ramie fibres are composed of small fibrils, the weakness caused by the "de-gumming" operation arises from the original fibres being broken up or separated into these smaller fibrils. I have it on the authority of Mr. Henry Ferguson, of Phoenix Mills, Brighouse, Yorkshire,—an expert possessing exceptional knowledge of ramie and vegetable fibres generally, and of their practical manipulation "from the field to the loom,"—that, whatever may be said to the contrary, the safest and most economical means of freeing the fibre from the "gum," pectic, and other bodies with least injury to its quality is the skilled use of hot alkali solutions in the first stage of operations. Those authorities who contend that the best practical results would be obtained with ramie fibre by spinning it in a condition "more nearly that in which it occurs in the plant," are reminded by an experienced spinner like Mr. Ferguson that the fibre must be first treated chemically "in order to stand the test of spinning"; in other words, the fibre in its natural condition cannot be advantageously employed for the finer textile purposes. It may be here incidentally mentioned that Mr. Ferguson has, after many years' work, succeeded in perfecting a rapid chemical means of almost

simultaneously de-barking and de-gumming the ramie ribbons without the aid of softening machinery, and so preparing the fibre as "to fit it for all the branches through which it has to be put in order to yield a strong and even yarn." This important invention is applicable to other vegetable fibres besides the one under notice.

According to Nodin and Brettoneau, the average composition of ramie stalks after de-gumming and drying may be stated as follows:—

Textile fibre	30 per cent
Wood	55 "
Bark, &c.	15 "

Dr. Collyer, by prolonged digestion in a simple soap solution, obtained 150 lbs. of pure fibre from a ton of green stalks. In Assam it has been found possible to obtain two tons of fibre per acre—from four successive crops grown in one season. The proportion of gum, pectic substances, and bark in the dried ribbons I found to vary from 20 to 30 per cent. But the proximate analyses of the ribbons I have been able to make are in some important respects imperfect, and I therefore refrain from quoting them, contenting myself with the hope that time and opportunity may enable me to hereafter deal exhaustively with that part of the subject.

Before passing on to treat of the ramie crop and the likely effect of its continuous growth and removal on the plant-food resources of the soil, I may briefly mention certain facts regarding the wild or uncultivated variety of the plant. It is met with in abundance in Japan, Java, South America, the Philippines, and other countries, and is generally believed to yield fibre of inferior quality, but Mr. Ferguson informs me that, working on a manufacturing scale with "brown ribbons" from South America, Assam, and elsewhere, he obtained an average return of about 70 per cent of pure fibre which was in every respect equal to the product given by the cultivated plant; indeed, it could be spun with cocoon silk in the proportion of one-third of the latter to two-thirds of the fibre, yielding a fabric which even experts could not easily distinguish from pure silk, and which was found to take the same dyes as the latter. Fibre which I had myself prepared from wild ramie I found to possess practically the same strength, dye-taking power, lustre, and other qualities as the product separated by the same means from the bark of the cultivated plant.

It may be interesting to quote the following analysis of the dried stalks and leaves of wild ramie from Japan:—

	Stalks.	Leaves.
Ash	4'94	18'72
Nitrogen	0'49	1'06
The ash contained—		
Potash	32'07	4'11
Soda	10'01	0'90
Lime	20'07	34'21
Magnesia	8'27	6'51
Phosphoric acid	14'48	4'81
Sulphuric acid	3'11	1'84
Silica	3'41	42'50
Chlorine (Cl ₂ —O = 55)	2'46	1'03

The ash of a whole plant (*B. calophleba*), indigenous to Lord Howe's Island, and sent to me by the late Baron Sir Ferdinand von Mueller, F.R.S., had the following composition:—

Ash	8'87 per cent
Nitrogen	1'65 "
The ash contained—	
Potash	5'57 "
Soda	2'08 "
Lime	33'05 "
Magnesia	7'09 "
Ferric oxide	1'83 "
Phosphoric acid	5'75 "
Sulphuric acid	2'07 "
Silica	36'98 "
Chlorine (Cl ₂ —O = 55)	2'45 "

It would not, of course, be wise to infer too much from these analyses, but, if anything, they would indicate that, at all events, the wild varieties of *Bomheria* contain an appreciably lower percentage of nitrogen and a generally similar quality of ash when compared with the cultivated plant. However, be this as it may, I quote the figures for what they may be worth, and I do so because I am unaware that any other analysis of the uncultivated plant is on record.

I have during the last five years accumulated a mass of analytical data concerning the composition of the ramie plant and its parts, but I cannot very well utilise it on this occasion, except in a general way, and that only so far as it throws light on the chemical effects likely to be produced on the soil by the continuous removal of crops from the same land.

In the following statement are arranged the analyses of the different parts of the cultivated ramie plant grown in Algeria, South America, Assam, Japan, Java, and England (Kew Gardens):—

- I. Stalks from which the bark had been stripped.
- II. Bark with fibre, gum, &c. (ribbons).
- III. Leaves.

I.
(Six Analyses).

	Minimum.	Maximum.	Average.
Total ash	3'10	3'49	3'25
Total nitrogen	0'81	0'92	0'82
The ash contained—			
Potash	36'88	38'08	37'58
Soda	9'01	16'00	10'86
Lime	16'07	17'98	17'52
Magnesia	10'63	11'20	10'67
Phosphoric acid	16'13	16'51	16'39
Sulphuric acid	2'62	3'78	3'51
Silica	1'29	2'15	1'81
Chlorine (Cl ₂ —O = 55)	1'93	2'19	2'03

II.
(Eight Analyses).

	Minimum.	Maximum.	Average.
Total ash	1'69	2'05	1'75
Total nitrogen	1'20	1'35	1'28
Ash contained—			
Potash	30'98	33'68	32'57
Soda	8'54	9'97	8'01
Lime	20'93	22'59	22'66
Magnesia	10'06	11'64	11'33
Phosphoric acid	10'88	13'04	12'57
Sulphuric acid	3'90	4'08	3'96
Chlorine	2'87	3'28	2'98
Silica	6'04	7'31	6'27

III.
(Four Analyses).

	Minimum.	Maximum.	Average.
Total ash	19'80	20'69	20'28
Total nitrogen	2'48	2'85	2'65
The ash contained—			
Potash	3'86	4'57	4'21
Soda	0'81	2'94	2'54
Lime	32'56	35'55	34'73
Magnesia	5'08	8'69	6'78
Phosphoric acid	5'09	5'85	5'27
Sulphuric acid	1'17	2'30	2'13
Silica	40'91	43'72	43'04
Chlorine	3'08	5'71	3'64

These results all refer to dried samples. In newly-cut stalks and leaves from the Royal Gardens at Kew, I found 90'86 and 90'59 per cent of water respectively.

The ramie plant thrives in a wide variety of soils, but best in rich friable loams containing humus, and preferably in localities where irrigation can be occasionally resorted

to. Under these conditions it ordinarily gives three crops a year, and in some countries—California, for example—it yields four. The presence of salt in the soil appears to be beneficial to ramie; indeed, Professor Hilgard says it is one of the very few crops which will prosper on "alkaline" land. On account of their richness in albumenoids, the leaves when green are very much liked by cattle, and for the same reason they can, according to Professor Dyer, F.R.S., be turned to account in feeding silk-worms.

Ramie is what may be termed a somewhat exhausting crop—drawing from the soil large quantities of plant food. Thus, a yield of nine tons of dried stalks and four tons of foliage would remove from an acre about 320 lbs. of nitrogen, 230 lbs. of potash, 140 lbs. of phosphoric acid, and no less than 600 lbs. of lime. An annual drain of this kind could not but ultimately tell upon the resources of even a naturally rich soil, and render it needful to resort to manuring in order to maintain fertility.

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A NEW SEPARATION OF THORIUM FROM CERIUM, LANTHANUM, AND DIDYMIUM, AND ITS APPLICATION TO THE ANALYSIS OF MONAZITE.*

By FLOYD J. METZGER.

(Concluded from p. 230).

Effect of Salts with Acetic Radicals on the Precipitation of Thorium by Fumaric Acid.

A VERY peculiar effect is noticed when an acetate is added to a solution of the rare earths (cerium, lanthanum, didymium, &c.) which has already had added to it some fumaric acid. The results are these:—

Take a solution of cerium (lanthanum or didymium) in 40 per cent alcohol and add to it a solution of fumaric acid; nothing results. If, now, one or two drops of ammonium acetate is added to this mixture of cerium and fumaric acid, the cerium is precipitated completely. Then if to this solution containing the precipitate an excess of ammonium acetate is added, the whole re-dissolves and leaves a clear solution. Cerium (lanthanum or didymium) is not precipitated by ammonium acetate alone. Ammonium fumarate precipitates cerium (lanthanum or didymium) insoluble in excess of precipitant. Add to cerium (lanthanum or didymium) dissolved in 40 per cent alcohol, fumaric acid, and then one or two drops of acetic acid, and nothing results; but then by adding to this one or two drops of ammonium acetate, the cerium is thrown down quantitatively as before. Thorium differs from cerium, lanthanum, and didymium in that the precipitate formed by fumaric acid is only partially soluble in an excess of ammonium acetate. Sodium acetate has the same effect as ammonium acetate.

The exact reaction in the above experiments has not yet been studied, but it is quite evident that salts of acetic acid must be avoided in the separation of thorium from cerium, lanthanum, and didymium by fumaric acid.

The Effect of other Metals on the Separation.

It was very desirable to know the effect of other metals on the precipitation of thorium by fumaric acid, so all the metals available were tried. The solutions in all cases (where such solutions could be obtained) were made in 40 per cent alcohol and the fumaric acid added. The following salts were tried:—

CuSO_4 ; AgNO_3 ; AuCl_3 (acid); MgCl_2 ; CaCl_2 ; $\text{Sr}(\text{NO}_3)_2$; BaCl_2 ; ZnSO_4 ; CdCl_2 ; $\text{Hg}_2(\text{NO}_3)_2$; $\text{Hg}(\text{NO}_3)_2$;

HgCl_2 ; H_3BO_3 ; $\text{Na}_2\text{B}_4\text{O}_7$; Al_2Cl_6 ; SnCl_4 (acid); SnCl_2 (acid); $\text{Pb}(\text{NO}_3)_2$; MnCl_2 ; Na_2HPO_4 ; As_2O_3 (slightly sol.); Sb (tatar emetic); $\text{Bi}(\text{NO}_3)_3$ (slightly sol.); $\text{UO}_2(\text{C}_2\text{H}_3\text{O}_2)_2$; VOCl_2 ; N_2WO_4 ; Fe_2Cl_6 ; FeSO_4 ; CoCl_2 ; NiCl_2 ; H_2PtCl_6 .

Of the above-named salts, AgNO_3 , $\text{Hg}_2(\text{NO}_3)_2$, and $\text{Hg}(\text{NO}_3)_2$ were the only ones which gave any precipitation. The precipitation of mercurous nitrate appears quantitative as well as the precipitation of mercuric nitrate, while mercuric chloride gives no precipitate whatever. The precipitation of silver nitrate is only a partial one.

Among the rare earths obtainable were yttrium, samarium, gadolinium, erbium, and zirconium, which, dissolved in 40 per cent alcohol, gave with fumaric acid:—

Yttrium gives no precipitate cold or hot. Samarium gives no precipitate cold or hot. Gadolinium gives no precipitate cold or hot. Erbium gives a very slight precipitate in the cold, which seems to increase somewhat on heating. Only a very small fraction, however, is thrown down. Zirconium gives a quantitative precipitation on heating.

The Composition of the Precipitate formed by Thorium and Fumaric Acid.

To determine the composition of the thorium fumarate, a quantity of pure salt was prepared by precipitating the purified thorium solution with fumaric acid, washing very thoroughly with 40 per cent alcohol, and drying. The salt was first dried at 105°C . in an air-bath, and then to determine the point at which the salt was decomposed, the temperature was gradually raised, causing a gradual loss in weight until at 202° to 205°C . it became practically constant, and no indications of decomposition were evident even at 245° to 247°C . This dried salt, however, re-absorbed moisture to a considerable extent when exposed to the air.

In order to obtain concordant results, the water was first driven off at 105°C ., collected, and weighed as atmospheric moisture. For this purpose a large tin can, provided with a thermometer, was placed upon the combustion furnace, and the tube passed through in the reverse position. Air was passed through the tube and the temperature maintained at 105° for four hours; the absorption tube was weighed, the can removed, and the combustion made in a current of oxygen. The water obtained in this was calculated as water of composition. The residue left in the boat should be thoria, and was weighed as such.

A number of combustions made in the above manner gave no constant results for hydrogen, while the thorium-carbon ratio was practically 1:4, as is seen from these results:—

	ThO_2	CO_2	Ratio.
I... ..	0.1319	0.0883	1:4.01
II... ..	0.1606	0.1086	1:4.05

The above ratios were calculated from the thoria just after removal from the furnace. After the combustion had been completed and the boat (containing the thoria) weighed, it was placed in a platinum tube and heated with a blast-lamp for ten or fifteen minutes. White fumes were given off, and the thoria decreased in weight to the extent of several milligrams. These fumes do not condense, but if made to pass through alcohol they are partially absorbed, and from this solution a very slight precipitate can be thrown down with ammonia. The losses were:—

0.1606 grm. thoria lost on ignition	0.0039 grm.
0.1147 grm. thoria lost on ignition	0.0036 grm.
0.1403 grm. thoria lost on ignition	0.0053 grm.
0.1641 grm. thoria lost on ignition	0.0054 grm.

Being under the impression that this loss was incurred by the co-precipitation (by fumaric acid) with thorium, of some compound which on ignition with the blast yielded a volatile oxide, it was thought advisable to prepare a

* Contribution from the Havemeyer Laboratories, Columbia University. Read at the meeting of the New York Section of the American Chemical Society. From the *Journal of the American Chemical Society*, xiv., No. 10.

quantity of thorium fumarate from thorium which had been precipitated with fumaric acid and thoroughly ignited. This was done in the following way:—The pure thorium solution was precipitated in the usual way with fumaric acid, and ignited for a period of one and one half hours in the strongest heat that could be obtained from a blast-lamp. The thorium was again brought back into solution after many evaporations with concentrated sulphuric acid, precipitated with ammonia, filtered, re-dissolved in nitric acid, evaporated to dryness, taken up in water, and re-precipitated with fumaric acid, thoroughly washed, and dried.

Combustions were then made on this compound as before:—

	ThO ₂	CO ₂	Ratio.	Loss on ignition.
I. ..	0.1870	0.1270	1 : 4.07	0.0066
II. ..	0.2434	—	—	0.0073

The same white fumes were driven off on ignition with the blast, causing a corresponding decrease in the weight of thorium left after combustion.

To learn whether the white fumes were a carbon-containing compound or not, the platinum tube was connected to the combustion furnace, and then heated with a blast. No increase in weight was obtained in the soda-lime tube, showing the absence of carbon, but an increase, slightly greater than the loss in weight of thorium, was found in the water-absorption tube (pumice and sulphuric acid).

To find out whether the fumaric acid employed was pure and free from any non-volatile substance, several portions were burned at a gentle heat in a Bunsen flame; no residue was left. A combustion of the acid was also made, giving:—

	Calculated.	Found.
H.	3.44	3.42
C... .. .	41.38	41.302

The fumaric acid was pure, and the white fumes obtained by the ignition of the thorium fumarate are as yet unaccounted for. These white fumes, however, will be made the subject of a separate investigation. The only conclusion to be drawn from the above work is that the thorium and fumaric acid react molecule for molecule. It is not supposed that the unsaturated valencies in the fumaric acid play any part in the reaction, but rather that fumaric acid is of just the proper strength to throw out the very feeble basic thorium.

Monazite Analysis.

To test the application of the fumaric acid to the analysis of monazite sand, three samples were procured: two Brazil sands and one from North Carolina,* and to check the results obtained, two of the best known methods, the thiosulphate and ammonium oxalate separations, together with a third,—a combination of the thiosulphate and oxalate,—were employed.

A description of the methods used is as follows:—

Ammonium Oxalate Separation.—About 1 gm. of the sand, ground to an impalpable powder, was weighed out into a platinum vessel, covered over with 15 to 20 c.c. concentrated sulphuric acid, and evaporated on an asbestos board until fumes were no longer driven off. More sulphuric acid was added, and this repeated several times until the conversion of the phosphates into sulphates was complete.

This mass was then projected in very small quantities into about 700 c.c. water at 0° C. with constant agitation the temperature was not allowed to rise above 2° C.). This was allowed to stand several hours with frequent stirring, was filtered and washed.† The filtrate was then nearly neutralised with dilute ammonia (1 : 20) and 50 c.c. of a cold saturated solution of oxalic acid added (for each

gm. of ore taken), stirred well, and allowed to stand. After the heavy white precipitate had settled completely, the solution was filtered, washed, and the precipitate washed into a 400 c.c. beaker, using a cold saturated solution of ammonium oxalate for the purpose. After making the volume of ammonium oxalate equal to about 50 c.c. the beaker was placed on a water-bath, covered with a watch-glass, and digested for one and one-half hours with occasional stirring. This was then diluted to 300 c.c., and allowed to stand over night, filtered (filtrate A), and washed. The residue was again subjected to the treatment with ammonium oxalate, &c., and filtered (filtrate B).

Filtrates A and B were combined and precipitated by a large excess of ammonia, heated to boiling, filtered, and the thorium hydroxide dissolved off the filter in hot dilute nitric acid; evaporated to dryness, the residue taken up in water, and precipitated with oxalic acid. This precipitate of thorium oxalate, which still contained some impurity, was again treated with ammonium oxalate as before (one treatment), precipitated with an excess of ammonia, converted into the oxalate, ignited, and weighed as thorium dioxide.

Thiosulphate Method.—The mineral was decomposed and brought into solution as before, and precipitated with oxalic acid. The precipitate of oxalates was washed into a beaker and treated with a strong solution of caustic potash (about 25 c.c.); heated to boiling (this converts the oxalates into hydroxides, which are then quite soluble in acids), diluted, and filtered; washed thoroughly, and dissolved off the filter in hot dilute hydrochloric acid (1 : 1); evaporated to dryness to free from acid, taken up in 75 to 100 c.c. water, and 15 c.c. of a saturated solution of sodium thiosulphate added, and heated to boiling (this precipitates nearly all the thorium together with a trace of impurity and considerable sulphur); filtered (precipitate A), and the filter set aside for subsequent filtration. The filtrate was precipitated by an excess of ammonia, filtered, washed, dissolved in hydrochloric acid, evaporated to dryness, taken up in water, and re-precipitated with thiosulphate as before. This was filtered through the paper containing precipitate A. The precipitation with thiosulphate was repeated, in the successive filtrates, as long as a precipitate was obtained (usually a third precipitation extracts the thorium completely).

The combined precipitates of thorium thiosulphate were washed completely, then dried and ignited. The ignited mass was fused several times with potassium bisulphate, taken up in water and a few drops of hydrochloric acid, and precipitated with oxalic acid. These oxalates were converted into the hydroxides as before, dissolved in hydrochloric acid, evaporated to dryness, taken up in water, and re-precipitated with sodium thiosulphate; filtered, washed, dried, and ignited. This was again fused with bisulphate, dissolved in water and a few drops of hydrochloric acid, and precipitated with oxalic acid; filtered, washed, ignited, and weighed.

It was necessary to fuse the thorium several times with bisulphate to get it all into solution.

Combustion Method.—Inasmuch as the methods just described are very lengthy and involve very troublesome processes, such as the repeated digestion of a large bulk of oxalates with ammonium oxalate, or the repeated fusions necessary in the thiosulphate method, it was found very convenient to make a combination of the two. Another point, well worth mentioning, and shortening the process greatly, is the conversion of the thorium thiosulphate back into the nitrate without ignition and repeated fusions. This is very easily and quickly done by washing the precipitate of thorium thiosulphate into a beaker with water, adding 20 to 25 c.c. of a strong solution of caustic potash, and heating to boiling. The thorium is converted completely into the hydroxide, while at the same time any free sulphur is dissolved by the caustic potash. The mass then is simply brought to boiling, diluted, filtered, washed, and dissolved off the filter in dilute nitric acid.

* It was through the kindness of Professor Baskerville that we secured this sample of North Carolina sand.

† Preliminary experiments showed that no metals precipitable by hydrogen sulphide were present, so this part of the process was omitted.

As has been noticed, the same reagent (caustic potash) serves for the conversion of the oxalates into hydroxides, which are then easily converted into the nitrates.

The combination method is as follows:—

Decompose the mineral as usual, and precipitate the rare earths as oxalates; filter, and wash the oxalates into a beaker; add 20 to 25 c.c. of a strong solution of caustic potash, and heat to boiling; dilute, filter, and wash. Dissolve the hydroxides off the filter with hot dilute hydrochloric acid (1:1), and evaporate to dryness on a water-bath. Take up with water, and add 25 to 30 c.c. of a saturated solution of sodium thiosulphate; heat to boiling, filter, and wash (precipitate A), and set the filter aside for subsequent filtration. Precipitate the filtrate with an excess of ammonia, filter, wash, dissolve off the filter with hot dilute hydrochloric acid, and evaporate to dryness. Take up with water, and re-precipitate with thiosulphate as before. Filter this precipitate through the paper containing precipitate A. Wash, then wash into a beaker, and add 20 to 25 c.c. of a strong solution of caustic potash, and heat to boiling. Dilute, filter, and wash. Then dissolve off the filter in warm dilute nitric acid, and evaporate to dryness. Re-dissolve in water, and precipitate with oxalic acid; filter, wash, and rinse into a beaker, using a cold saturated solution of ammonium oxalate for the purpose (about 100 c.c.).* Digest this on a water-bath for one and one-half hours (covered with a watch-glass), dilute to 300 c.c., and allow to stand over night. (The undissolved residue of cerium, lanthanum, didymium, &c., is so slight that it need not be re-treated). Filter, and precipitate the thorium from the filtrate with a large excess of ammonia. Filter, dissolve in dilute nitric acid, evaporate to dryness, take up in water, and precipitate the thorium from this with oxalic acid. Filter, wash, ignite, and weigh.

The above method gives very satisfactory results, and yields a white oxide of thorium.

Fumaric Acid Method.—The three methods just described are very long and tedious. In many instances the solutions must be allowed to stand over night, and the precipitations repeated several times to insure a complete separation. The minimum time required to execute any one of these methods is from five to six days.

The method about to be described has the great advantage of being extremely short, requiring only about one and one-half days after the decomposition of the mineral, and gives accurate results. The method is as follows:—Decompose about 1 grm. of mineral as before, and precipitate the oxalates; wash, then rinse into a beaker, and add 20 to 25 c.c. of a strong solution of caustic potash; heat to boiling, dilute, filter, and wash. Dissolve off the filter in warm dilute nitric acid (1:1), and evaporate to dryness on a water-bath. Take up in 50 c.c. water, then add alcohol and water in such proportions as to make the solution 40 per cent alcohol (dilution = about 200 c.c.). Then add 20 to 25 c.c. of fumaric acid (0.1 grm. per 10 c.c.), and heat to boiling. Filter, while still hot, through a long-stem funnel (or by suction), wash several times with hot 40 per cent alcohol, then return precipitate and paper to the beaker, and add 25 to 30 c.c. of dilute hydrochloric acid (1:1); heat to boiling, dilute a very little (keep down the volume as much as possible), and filter off the paper. Wash the paper several times, then evaporate to dryness on a water-bath, shaking from time to time, and washing with a few drops of water to prevent the residue from clinging to the sides of the beaker. Add about 50 c.c. water (while still on the water-bath, and stir the residue loose from the bottom with a rubber-capped rod. (The carbonaceous matter from the partly decomposed fumaric acid, &c., does not interfere). Add alcohol and water to make the solution 40 per cent (dilution about 150 c.c.), then add about 10 c.c. fumaric acid, and heat to

boiling. Filter through a long-stem funnel, wash with hot 40 per cent alcohol, ignite (without previous drying) in a platinum crucible, and weigh the thorium dioxide. In this method hydrogen sulphide need not be passed through the solution before precipitation with oxalic acid, for, as has been shown, none of the metals precipitable by hydrogen sulphide interfere.

A comparison of the analyses of the three samples of sand, made by the methods described, is given in the following table. Results are given in percentages of thorium:—

	Methods.			
	Fumaric.	Oxalate.	Thiosulphate.	Combination.
Brazil (a) . .	{ 2'41 2'55	{ 2'77 2'03	{ 2'11 —	{ 2'48 —
North Carolina	{ 3'95 4'02 3'92	{ — — —	{ — — —	{ 4'03 3'76 —
Brazil (b) . .	{ 2'51 2'47	{ — —	{ — —	{ 2'42 2'56

Conclusions.*

1. A saturated solution of fumaric acid in 40 per cent alcohol precipitates thorium completely from neutral solutions, to which, 40 per cent of their volume of alcohol has been added, while under these conditions the only other metals that give precipitation are zirconium, erbium, silver, and mercury.

2. This precipitation serves as an accurate and rapid separation of thorium from the other earths in monazite, and by its use the thorium can be determined in about one-third the time required by the methods now in use, and with equal, if not greater, accuracy.

3. As the thorium-carbon ratio in thorium fumarate is 1:4, thorium and fumaric acid react molecule for molecule.

4. By the aid of a blast-lamp, white vapours are driven off from the thorium left in the boat after the combustion of thorium fumarate in a stream of oxygen (a fact as yet not explained).

5. Rare-earth oxalates and thorium thiosulphate are completely converted into the hydroxides by heating to boiling in a strong solution of potassium hydroxide, giving a convenient method for the conversion of those salts into the nitrates.

This investigation was carried out under the direction of Professor Edmund H. Miller, and I wish here to express my most sincere thanks to him for his kind interest and encouragement in the work, and for the valuable assistance rendered.

PROCEEDINGS OF SOCIETIES.

MANCHESTER LITERARY AND PHILOSOPHICAL SOCIETY.

Ordinary Meeting, November 4, 1902.

Mr. CHARLES BAILEY, President, in the Chair.

A PAPER ON "*The Action of Alkalis on Glass and on Paraffin*" was read by Mr. FRANCIS JONES, M.Sc., F.C.S., F.R.S.E.

He pointed out that, while it is generally acknowledged that alkalis in course of time act on glass, there is considerable difference of opinion among chemists as to whether this action interferes with the well-known test for carbon dioxide in air, generally known as Pettenkofer's, but which was first described by Dalton in a paper read before this Society in 1802. Solutions of lime, strontia, and baryta of known strength were left in glass bottles at the ordinary temperature for several months, and the strength of each was ascertained from time to time. It was found that the lime-water lost strength more rapidly

* By this process the oxalate to be digested with ammonium oxalate is almost pure thorium oxalate and goes into solution very easily. Usually only the faintest residue of impurities remains after one digestion.

than the others, and that baryta could be kept in glass bottles for a period of twenty months without suffering any material loss in strength. Similar solutions were left in contact with finely divided silica and with powdered glass, and again it was found that lime-water acted on these bodies more rapidly than the other two. The action on glass bottles, however, is not so rapid as to prevent any three of these alkaline solutions being used for Pettenkofer's test. It has been suggested that bottles used for this test should be coated with paraffin wax, to prevent the contact of the alkaline liquid with the glass, but the author shows that lime, strontia, and baryta lose strength in contact with paraffin, the action of baryta being much more energetic than that of either lime or strontia. Some baryta solution in contact with paraffin for five months was very nearly neutral at the end of that period. Consequently, the storing of standard baryta solutions in paraffined bottles is quite inadmissible.

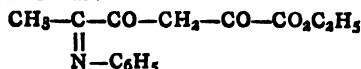
CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxv., No. 16, October 20, 1902.

The Formation of Liquid Drops and Tate's Law.—Ph. A. Guye and P. Louis Perrot.—The authors endeavour to explain the complex phenomena taking place during the formation of drops issuing from the extremity of cylindrical tubes with capillary bore. They also examine the successive forms each drop takes before its fall. A photographic record gives evidence quite independent of retinal illusions. They find once again that the classic relations of Tate do not correspond to the actual facts, for the rupture of the drop does not take place when it has a diameter nearly that of the tube; also that the fall of the drop preceded by the formation of a thread ought rather to be compared to the phenomena of rupture of metallic threads under a dragging force, and that consequently the rigidity of liquids ought to play a part in the formation of drops, which part still remains to be investigated.

Derivatives of Pyruvyl Pyruvic Ether. Stereoisomeric Hydrazones.—L. J. Simon.—The consecutive actions of aniline and strong sulphuric acid on ethyl pyruvate lead to the preparation of a body to which the author assigns the formula—



and which therefore may be considered as a phenylimine derivative of pyruvyl pyruvic ether. In order to prove the ketonic nature of this compound he submits it to the action of the characteristic reagents for such substances—first, to that of phenylhydrazine, and then to that of concentrated sulphuric acid.

Action of Mixed Organo-magnesium Compounds on the Ethers of the Ketonic Acids.—V. Grignard.—The author has already shown that the β -ketonic ethers generally give with organo-magnesium compounds abnormal reactions in which above all the presence of the enolic form is predominant. Such is not the case with the other ketonic ethers, which are capable of reacting normally by their two-functional grouping. But, as it may be predicted, these two groups do not present the same reaction velocity; carbonyl reacts always before carboxyl, if the method allows of the tertiary alcoholic acids being obtained or the bitertiary glycols, which happens when one molecule or three molecules of the organo-magnesium compound acts on one molecule of the ketonic ether. The author is mainly concerned with the synthesis of acid

alcohols. In his experiments, at each instant it was necessary to avoid the presence of an excess of the organo-metallic compound, so as to prevent it acting on the carboxalkyl, and to do this it was essential to allow the magnesium compounds to fall little by little into the ketonic ether. His experiments were performed on isoamyl pyruvate, ethyl phenyl glyoxylate, ethyl levulate, and ethyl acetyl succinate. A list of results is given.

MISCELLANEOUS.

Some Crystallised Alloys of Aluminium.—O. Brunck.—*Copper and Aluminium*, Cu_4Al_9 .—Equal weights of the two metals are melted together, and very fragile, crystalline, white ingots are obtained. By decanting before complete solidification magnificent lance-like crystals can be obtained; density = 4.118. These crystals are slightly attacked by nitric acid, partially by hydrochloric acid, and completely by aqua regia. *Iron and Aluminium*, FeAl_3 .—One part of iron is melted with three parts of aluminium, and then treated with 2 per cent hydrochloric acid. Pointed crystals of an iron-grey colour are obtained; soluble in strong acids; density = 3.734. *Nickel and Aluminium*, NiAl_3 .—This occurs in crystals similar to the last, and they have the same colour as nickel; density = 3.681. *Cobalt and Aluminium*, $\text{Co}_3\text{Al}_{13}$.—Crystals of a similar appearance; density = 3.492. *Manganese and Aluminium*, Mn_2Al_7 .—Under a layer of common salt, we melt one part of manganese and six parts of aluminium, and then treat with 2 per cent hydrochloric acid. White crystals are formed having the appearance of tin. *Platinum and Aluminium*, $\text{Pt}_3\text{Al}_{10}$.—This compound requires a prolonged fusion. Rather indistinct crystals are formed of a bronze colour, decomposed by concentrated hydrochloric acid; density = 6.688.—*Berichte*, vol. xxxiv., p. 2733.

Estimation of Sulphuric Acid in Mineral Waters.—L. W. Winkler.—Sulphuric acid is precipitated by a hydrochloric solution of chromate of barium. The excess of chromate being precipitated in its turn by neutralisation, the chromic acid displaced by the sulphuric acid is determined colorimetrically, by comparison with a titrated solution of chromate of potassium. The solubility of the chromate of barium in a neutral solution not being negligible, the necessary correction is determined by the equality of the tints between a known solution of chromate of barium and the equivalent typical solution of the salt of potassium. The method of procedure is as follows:—To 150 or 200 c.c. of the water under examination, we add 5 or 10 drops of fuming hydrochloric acid, then 0.1 to 0.2 gm. of chromate of barium; boil, neutralise with soda after cooling, then filter. Take 100 c.c. of the filtrate, and, at the same time, 100 c.c. of distilled water treated with a few drops of soda; pour into this latter solution the solution of chromate of potassium until the tint is the same as that of the former. The same operation being carried out on a titrated solution of sulphate of potassium containing 1 gm. of SO_3 per litre, the value in SO_3 of the water in question is easily calculated. The possible error is from 1 to 2 mgrms. per litre, and the figures thus obtained with mineral waters agree with the results obtained by the gravimetric method.—*Zeit. Anal. Chim.*, vol. xl., p. 465.

Simultaneous Estimation of Cyanides and Cyanates.—Ernst Victor.—This method depends on the solubility of cyanate of silver in dilute nitric acid. Two portions of 10 c.c. each of the 10 per cent mixture are treated with a known excess of decinormal nitrate of silver. One portion made up to 100 c.c. is filtered, and the excess of silver titrated in an aliquot part of the filtrate. The other portion is treated first with dilute nitric acid, made up to 100 c.c., and the excess of silver determined in the same manner. The difference between these two titrations gives the amount of cyanate contained

in the sample. The author criticises the propositions put forward by Feldtmann and Buttel with regard to the same estimation. Unlike them, he did not find it necessary to work at a temperature of 0° to precipitate the cyanate of silver at the same time as the cyanide. At a temperature of 25° it is only after the lapse of several hours that the decomposition of the cyanate into ammonia and bicarbonate of potassium is noticed, and not the transposition, $\text{OCNK} + 2\text{H}_2\text{O} = \text{CO}_2\text{KH} + \text{NH}_3$.—*Zeit. Anal. Chim.*, vol. xl., p. 462.

NOTES AND QUERIES.

* * Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Pure Cyanide.—We are in the market for large quantities of chemically pure cyanide, but are not informed as to who the manufacturers are in England and Scotland. We would be under obligations if you would furnish us with the names of said manufacturers.—H. P. D.

Action of Red Light.—As a constant reader of your valuable paper, I shall esteem it a favour if any of your scientific correspondents can inform me what is the action of the red rays of light on the hair, and what authority is there for supposing that they have a beneficial effect and promote the growth?—F. H. BAILY.

MEETINGS FOR THE WEEK.

WEDNESDAY, 19th.—Chemical, 5.30. "The Dynamic Isomerism of Thiourea and Ammonium Thiocyanate," by J. E. Reynold and E. A. Werner. "Isomeric partially Racemic Salts containing Quinquevalent Nitrogen—Part VIII., Re-solution of the Hydriodamine Bromocamphor Sulphonates," and "Isomeric Compounds of the Type $\text{NR}_1\text{R}_2\text{H}$," by F. S. Kipping. "The Synthesis of α -Dimethylglutaric Acid, of β -Hydroxy- α -Dimethylglutaric Acid, and of the *cis*- and *trans*-modifications of α -Dimethylglutaconic Acid," by W. H. Perkin, jun., and Miss E. Smith. "A Reaction of some Phenolic Colouring-matters" (Part II.), by A. G. Perkin and C. R. Wilson. "The Vapour-pressures and Boiling-points of Mixed Liquids" (Part II.), by S. Young and Miss E. C. Fortey. "The Vapour-pressures and Boiling-points of Mixed Liquids" (Part III.), and "Note on Mixtures of Constant Boiling-point," by S. Young. "Note on the Condensation-points of the Thorium and Radium Emanations," by E. Rutherford and F. Soddy. "The Oxime of Mesoxamide and some Allied Compounds—Part II., Di-substituted Derivatives," by Miss M. A. Whiteley.

Society of Arts, 8. Opening Meeting of the 149th Session. Inaugural Address by Sir William Henry Preece, K.C.B., F.R.S., Chairman of the Council.

Microscopical, 8. "An Electrical Method of taking Microscope Measurements," by Dr. Philip E. Shaw. There will also be Demonstrations on "The Microscope in Fossil Botany," by Dr. Dukinfield H. Scott, F.R.S., and on "An Apparatus for obtaining Monochromatic Light with the Mixed Jet," by Dr. Edmund J. Spilla.

In the Matter of the COMPANIES' ACTS, 1862 to 1900,

and
In the Matter of the MINERALS REDUCTION, LIMITED.

The CREDITORS of the above-named Company are required on or before the 10th day of December next to send in their names and addresses, and the particulars of their Debts or Claims, and the names of their Solicitors (if any), to the undersigned CHARLES WHINFIELD ASTON KEY, of a Metal Exchange Buildings, Lendenhall Avenue, London, E.C., the Liquidator of the said Company, and if so required in writing from the said Liquidator are by their Solicitors to come in and prove their Debts or Claims at such time and place as shall be specified in such notice, or in default thereof they will be excluded from the benefit of any distribution made before such Debts are proved.

C. W. ASTON KEY,
Liquidator.

a, Metal Exchange Buildings,
Lendenhall Avenue, London, E.C.,
November 7th, 1902.

INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

(Founded 1877, Incorporated by Royal Charter 1885).

NOTICE IS HEREBY GIVEN, that the NEXT EXAMINATIONS of the INSTITUTE OF CHEMISTRY, for persons desirous of qualifying themselves to be Public and Technical Analysts, will be held at 30, Bloomsbury Square, London, W.C., in JANUARY, 1903.

The Examinations are open only to Candidates who have complied with the Regulations.

The Intermediate Examination will occupy at least four days, commencing on TUESDAY, JANUARY 6th.

Examinations in the following Branches of the Final Examination for the Associateship (A.I.C.) will extend over at least four days, and may commence on either January 6th or 13th:—(a) Mineral Chemistry; (b) Metallurgical Chemistry; (c) Physical Chemistry; (d) Organic Chemistry; and (e) The Analysis of Food and Drugs, and of Water, including an Examination in Therapeutics, Pharmacology, and Microscopy.

(The Final Examination in Branch (f) Biological Chemistry is held in October each year).

Applications for admission to the January Examinations should be forwarded to the Registrar not later than TUESDAY, DECEMBER 23rd, 1902, on which day the List of Candidates will be closed.

By Order of the Council,

RICHARD B. PILCHER,

Registrar and Secretary.

30, Bloomsbury Square, London, W.C., October, 1902.

* "The Regulations for the Admission of Students, Associates, and Fellows of the Institute of Chemistry" may be obtained from Messrs. Blundell, Taylor, and Co., 173, Upper Thames Street, London, E.C., price One Shilling.

THE BARROW SALT CO., LIMITED, WALNEY, BARROW-IN-FURNESS.

Highly important SALE in ONE LOT, as a going concern, of the SALT WORKS, BRINE WELLS, RESERVOIRS and FILTER-BEDS, COTTAGES, &c., situate on Walney Island, near Barrow-in-Furness, together with the FIXED PLANT, MACHINERY, RAILWAYS, and GOODWILL of the above undertaking.

TO BE SOLD BY AUCTION by EDWARD RUSHTON, SON, and KENYON, at the Law Association's Rooms, Cook Street, LIVERPOOL, on THURSDAY, the 4th day of DECEMBER, 1902, at THREE o'clock in the afternoon, subject to conditions as set out in printed Particulars of Sale:—

ALL THOSE THE SALT WORKS, Brine Wells, Reservoirs, Filter-beds, Fixed Plant, &c., of the above Company, full particulars of which are given in Particulars of Sale.

Full descriptive particulars and Plans may be obtained from the AUCTIONEERS, 13, Norfolk Street, Manchester; Messrs. W. B. PRAT and Co., Chartered Accountants, 125, Ramadan Square, Barrow-in-Furness; or from Mr. C. F. PRESTON, Solicitor, 4, Lawson Street, Barrow-in-Furness.

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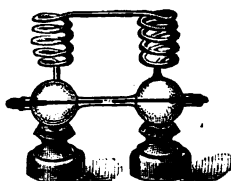
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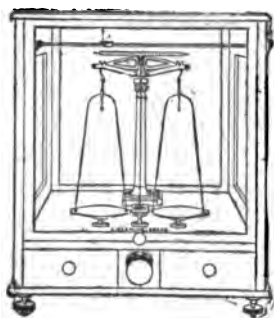
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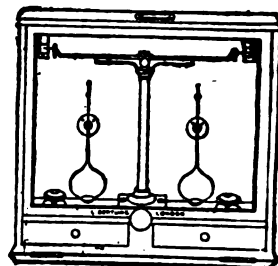
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Vol. LXXXVI., No. 2243.

SOME OBSERVATIONS ON THE FLUORESCENCE AND PHOSPHORESCENCE OF DIAMONDS, AND THEIR INFLUENCE ON THE PHOTOGRAPHIC PLATE.

By OTTO ROSENHEIM, Ph.D., F.C.S.

It has been observed by W. Marckwald (*Chem. Zeit.*, xxvi., 895), that the rays emitted by radioactive polonium possess the property of inducing fluorescence in diamonds. The present writer has examined a large number of diamonds of different origin (Cape, Brazil, British Guiana, India), which all exhibited this phenomenon. The intensity of the fluorescence apparently varied with the size and quality of the diamonds, but they could all be distinguished from other precious stones, such as ruby, emerald, zircon, topaz, opal, &c., by their emission of light under the influence of polonium in an absolutely dark room.* The so-called black diamonds or carbonados were not affected by polonium.

The rays emitted by the diamond under the influence of polonium affect not only the retina, but also the photographic plate. If in the absence of actinic light a diamond and a polonium rod† placed closely together, are brought into contact with a photographic plate, development after an exposure of two minutes' duration demonstrates the reduction of the bromide of silver not only along the polonium rod, but also underneath the diamond, the image produced in this case being much more striking and sharply defined. A similar effect is observed if the distance between the diamond and rod be increased to 10 m.m. or more.

This actinic activity of the diamond, like its visible fluorescence, is entirely dependent on the presence of the polonium, not persisting after the removal of the latter. Even after long exposure to polonium rays no induced radioactivity could be detected.

Although the rays emitted by polonium and those emitted by the diamond under its influence are qualitatively alike in their effect on the photographic plate, they differ in their absorbability by various substances. As is well known, the former are easily absorbed by tissue paper, celluloid, gutta-percha tissue, and glass, and exert no influence on the plate through these. On the other hand, the rays emitted by the diamond, under the influence of polonium, easily penetrate thin films of these substances or glass plates a few millimetres thick. This may be demonstrated by interposing between the sensitive plate and the diamond and polonium rod a sheet of paper, of gutta-percha tissue, or of celluloid. On development an impression of the diamond only is obtained. The same difference of effect is observed if the diamond and polonium rod are placed in contact with the "back" of the photographic glass plate or celluloid film, the polonium rays being absorbed completely by the glass or celluloid.

The observations described seemed to make it of interest to examine the effect on the photographic plate of phosphorescent diamonds. It has been known since R. Boyle first described this phenomenon in 1663 that certain diamonds, after exposure to strong sunlight, possess the power of sending out phosphorescent light. Quite recently the late J. H. Gladstone (*Brit. Assoc.*

Report, 1902; *CHEM. NEWS*, lxxxvi., 176), added another to the many instances of this phenomenon already known. The present writer has examined a great number of diamonds with regard to the possession of this property, making use, however, in the absence of strong sunlight of the actinic light emitted by burning magnesium ribbon. Of the diamonds thus examined only three were phosphorescent. With these an exposure of a few seconds to magnesium light was sufficient to produce phosphorescence as intense, judged by the eye, as the fluorescence produced by polonium. In spite of this the phosphorescence of none of these diamonds, produced by even a long exposure to the light, had the slightest effect on the photographic plate, even after a contact with the latter extending over 24 hours. It is interesting to note that of these three diamonds two were known with certainty to be of Brazilian origin, whilst the third was described as such.

The experiments described have shown:—

1. That all the diamonds examined fluoresce and affect the photographic plate under the influence of polonium.
2. That the rays emitted by the diamonds under these conditions differ from the polonium rays by their power of penetrating various media.
3. That certain diamonds phosphoresce after exposure to magnesium light, but their rays do not affect the photographic plate.

King's College, London,
November, 1902.

THE ULTRA-VIOLET SPARK-SPECTRA OF THE ELEMENTS.

By Prof. FRANZ EXNER and Dr. E. HASCHKE.

Radium.

The spark spectrum of radium has been investigated by Demarcay (*Comptes Rendus*, 1899, cxxix.; 1900, cxxxi.), Runge (*Ann. der. Phys.*, 1900, Bd. ii.), and Berndt (*Phys. Zeit.*, 1900-1901, Bd. ii.). Considering the difficulty of obtaining the material, and its want of uniformity according to the method of preparation employed, it is not surprising that the results of the several observers, as regards the identification of the lines, do not agree in many cases. The material which we had at our disposal we obtained as chloride from the Société des Produits Chimiques, in Paris, with an activity of 240. The number of the lines which we must in all probability ascribe to radium is seventeen.

λ	μ	
2512.46	1 +	Very faint in Ba.
2736.20	1 + br.	—
2813.60	1	Very faint in Ba.
16.25	2 + br.	—
31.98	1	—
77.10	1 +	Very faint in Ba.
2908.0	1 + br.	—
76.17	1 +	—
3079.97	2	Faint in Ba.
3126.53	1	Very faint in Ba.
3541.77	1 +	Very faint in Ba.
3649.33	2 +	Faint in Ba.
3814.62	3	—
61.77	3 + *	Faint in Ba.
3993.25	3 + †	—
4053.81	1 +	Very faint in Ba.
4682.41	1	—
4781.4	1 + br.	—

* This line does not belong to radium.

† This line is very close to a strong barium line.

The lines which we mark as occurring also in Ba were observed in a very strongly exposed spectrum of barium nitrate. Considering the relative intensity of the strong

* It hardly needs special mention that the phenomena of fluorescence and phosphorescence described can only be observed if the eyes have been "adapted" for some time to complete darkness.

† The commercial polonium-blamuth rod, prepared according to Marckwald, was used for these experiments.

barium lines in the radium preparation and in barium nitrate, it is difficult to believe that these lines belong to barium. We have therefore provisionally included them in the table of radium lines. As Runge remarks, some of the lines which Demarcay ascribes to radium certainly belong to barium. However, the lines 4627.4 and 4699.8, which are both missing in our radium spectrum, differ so much in wave-length from the neighbouring barium lines that they can hardly be identified with these. The line 4641.9 belongs to oxygen. Demarcay's five lines between 4340 and 4533 were invisible to us, as also to Runge. They appear, at least in part, to belong to Ti and Ca. Of the three lines which Runge specifies for radium, we could not find the one 4826.14, which Demarcay also has observed; Berndt, too, does not mention this. On the other hand, this observer gives a line at 2708.6, which none of the other observers have seen. Only the two lines 3814 and 4682 are common to all measurements; the latter has also been observed by Miethe (*Phys. Zeit.*, Bd. 2, Feb., 1901), when investigating radium with a new spark apparatus. A second line, $\lambda = 4348$, described by him as very strong, is not mentioned by any other observer.

Polonium.

There is still more uncertainty about the spectrum of polonium than about that of radium. Demarcay (communicated by P. Curie, *Comptes Rendus*, 1898, cxvii.) and Runge (*loc. cit.*) could find no lines. Berndt (*loc. cit.*) found fifteen, but it is very doubtful whether some of them belong to polonium. We have examined two different preparations: an older preparation by M. Curie (bismuth nitrate), with an activity of 5000; and a polonium-bismuth metal of activity 300, as supplied by the Société Centrale des Produits Chimiques. In the first we were able to discover a series of faint lines which we could not identify with any known lines. We quote them here for the sake of completeness, without wishing to affirm that they belong to polonium.

Bismuth-polonium Nitrate.

λ	λ
3289.49	2 Yb?
3944.90	2 + Ho?
4167.81	2

In the second preparation we found only Fe, Bi, As. We have only given the more strongly marked lines of the first preparation. They cannot be definitely ascribed to polonium; they do not in the least agree with those which Berndt has found. Of these last, the greater number certainly belong to impurities, which, as far as identification is possible, we denote in the following table of Berndt's lines by the annexed symbols:—

λ	λ	λ
2327.3 Fe	3168.2 Ti	3462.0 Ti
2661.9 Sn? Pb?	3191.2 Ti	3467.1 Cd
2977.0 Ru	3349.2 Ti	3821.9 Rh?
3152.2 Ti	3361.5 Ti	4007.7 Ti
3162.9 Ti	3342.0 Mn?*	4596.3 O

* The wave-length appears to be wrongly placed by a printer's error.

The lines given here do not agree with ours, so that we must say that up to the present no polonium lines have been established with certainty. — *Aus den Sitzungsberichten der Kaiserl. Akademie der Wissenschaften in Wien.*

The Concise Chemical Analysis Chart.—We have received a copy of this Chart from the publishers, Messrs. Jarrold and Sons, London, and can recommend it for the laboratory use of students. The Chart contains not only instructions for chemical analysis by the ordinary group method, but also a number of confirmatory tests by the dry way, in the flame, on charcoal, &c. It is arranged in a convenient form, and is more suitable for bench-work than a text-book as generally used.

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I. INTRODUCTION.

Historical.

In the year 1843 Mosander discovered a new rare earth, the pink-coloured terbium earth (the erbium earth of Berlin and subsequent investigators); this new earth was afterwards subjected to further examination by Bahr and Bunsen, as well as by Höglund.

Thirty-five years later, however, Marignac (*Arch. des Sciences Phys. et Nat.*, November 18, 1878) succeeded in proving that the erbium earth was not a single substance, but that, by partial decomposition of the molten nitrate, it might be resolved into two components—a pink-coloured earth for which the old name was retained, and a colourless earth which he called ytterbium.

Owing to lack of material, Marignac could not continue the investigation of this newly-discovered colourless earth; but its further examination was carried on without delay, since L. F. Nilson (*Öf. K. Vet. Ak. Förh. Stockholm*, 1879, No. 3) in the next year (1879) was able to issue a report on his experiments on the isolation of ytterbium. This author finally obtained an earth which in solution was completely free from all absorption-bands; its atomic weight remained constant at 132 (R^{10}O , i.e., at 174, calculating from the formula of the sesquioxide); this value was slightly higher than the number obtained by Marignac—130.8 (and 172.2 respectively).

In the same investigation Nilson was led to the discovery of scandium. After continued fractionating, the atomic weight of one portion of the material was found to be considerably lower. All these fractions were taken and purified. Further examination showed that they owed their diminished atomic weight to the admixture of a new substance—scandium.

In the following year Nilson published further details concerning scandium (*Öf. K. Vet. Ak. Förh. Stockholm*, 1881, No. 6, S. 16). He had by that time succeeded in obtaining 20 grms. of the rare earth. After careful purification and separation of the scandium, the atomic weight was fixed at 173.01 ($\text{O} = 16$); this number was not altered by further fractionating. The fact that Nilson had previously obtained a higher number, 174, is to be ascribed to the presence of traces of platinum, derived from the vessels in which the experiments were performed.

Besides the oxide, Nilson prepared the hydrate, nitrate, sulphate, acid selenite, and oxalate; the three last being also analysed. If we add that Thalén a short time afterwards described the spark-spectrum of ytterbium, we have stated all that is at present known of the metal. We may summarise our knowledge as follows:—Ytterbium is probably an elementary substance with atomic weight 173. It possesses at least one salt characteristic of trivalent metals, namely, the acid selenite. The salts have no absorption spectrum if the acid ion is colourless.

In consideration of the fact that up to the present very few reports on the characteristic properties of the metal have been published, I was induced to carry on further investigations, particularly as regards its compounds, which for the most part have not yet been described. This seemed especially desirable, since a great deal of work has recently been done on the elements most closely allied to it, e.g., gadolinium and praseodymium.

I was all the more impelled to carry on this research because fairly considerable quantities of the earth in question were to be obtained from the University Chemical Laboratory at Upsala, and this material was most kindly placed at my disposal. I take this opportunity of expressing my thanks to the Directors of the Institute, my father Professor P. T. Cleve, and Professor O. Pettersson, who

* From the *Zeitschrift für Anorganische Chemie*, xxii.

have rendered me assistance by giving me both material and valuable advice. The work was performed in the Chemical Institute of the Stockholm High School.

II. PREPARATION OF PURE MATERIAL. THE ELEMENTARY NATURE OF YTTERBIUM. ATOMIC WEIGHT AND VALENCY.

The earth was prepared from a mixture, amounting in weight to several kilograms, of ytterbium earths derived from many different minerals. Cerium oxides were previously removed by treatment with potassium sulphate, and the recovery of pure ytterbium then fell into two sections.

Firstly, the greater quantity of ytterbium was separated according to Marignac's well-known method. Nilson had already shown that this method was preferable to Bunsen's in the case in question. Crude ytterbium can be extracted from the remaining mixture comparatively quickly if the heating of the nitrate is stopped when the consistency of a fairly mobile fluid is reached, so long as yttrium and erbium are present in considerable quantity, but the heating must be increased in proportion as the mixture is richer in ytterbium.

As a rule each greater fraction was subjected to two successive partial decompositions, and those portions were purified which showed sufficient agreement when examined spectroscopically.

In other cases, especially for the separation of erbium and holmium, the method of partial decomposition of the nitrate is long and tedious, but it is useful if it is required to separate ytterbium from greater quantities of yttrium, holmium, and erbium in this order. After the strongly marked absorption-bands caused by the erbium had disappeared in the green region, the fused nitrate still showed for some time a violet colouration due to thulium. There still remained the lengthy task of completely purifying the ytterbium earth which had been freed from erbium; for only by repeated fractionation could the ytterbium be freed from thulium without excessive loss of material. Nilson also mentions the difficulty of getting rid of all the thulium.

In this way I obtained about 150 grms. of an earth the concentrated solutions of which were colourless. It was, of course, possible that scandium was still present. The whole quantity was therefore split up into new fractions, the atomic weights of which were determined in the ordinary way by converting the oxide into sulphate.

I found the following values for the first—least basic—fractions, in which the scandium must be present in greatest quantity.

(NOTE.—In this, as in all subsequent calculations, the atomic weights established by the Commission on Atomic Weights are employed).

I	..	..	..	..	..	173.3
2	..	..	..	..	..	172.8
3	..	..	..	..	..	172.1
4	..	..	..	..	..	172.9
5	..	..	..	..	..	172.3

Hence it follows immediately that scandium was not present to a perceptible extent.

The original material was now split up into five parts, and their atomic weights were ascertained:—

		Oxide. Grm.	Sulphate. Grm.	Atomic weight.
I.	..	0.3919	0.6319	172.15
II.	..	0.4793	0.7728	172.15
III.	..	0.3693	0.5963	171.4
IV.	..	0.6121	0.9867	172.23
V.	..	0.6842	1.1027	172.34

Since the atomic weights agreed so closely that the earth might be regarded as tolerably homogeneous, it was subjected to a last purification, in accordance with Nilson's directions (*Öf. K. Vet. Ak. Förh. Stockholm*, 1880, No. 6, p. 6), in order that any foreign matter which

had been introduced during the process of separation might be removed. In the first place, a small quantity of the platinum of the dishes always goes into solution. In order to ascertain the influence of this platinum on the atomic weight, all the platinum from Fraction III. above was removed by means of sulphuretted hydrogen, and the neutral solution after filtration was precipitated with oxalic acid (III a).

Ammonia still gave a small precipitate in the filtrate III. b. The atomic weights of the two fractions were:—

	Oxide. Grm.	Sulphate. Grm.	At. weight.	Colour of oxide.
III. a.	0.4204	0.6800	170.86	Faint brown.
III. b.	0.5739	0.9271	171.14	Yellowish brown.

Thus, before purification, the presence of platinum caused the value found to be too high by unity, or at the least by one-third.

When all the material had been similarly treated, the last traces of foreign earths were removed by decompositions of the nitrate. The highest atomic weights thus obtained were those of the three fractions—

Oxide. Grm.	Sulphate. Grms.	Atomic weight.
0.7791	1.2535	173.22
0.5190	0.8353	173.05
0.4905	0.7894	173.07
Mean		173.11

Further decomposition of these fractions did not lead to higher values.

The number 173.01 fixed by Nilson corresponds to 173.16 after correction for the atomic weights employed throughout these calculations, and thus agrees with my mean value given above, within the limits of experimental errors. Hence I saw no reason for further investigating the atomic weight or elementary character of ytterbium, but contented myself with confirming Nilson's conclusions on these points. The atomic weight 173.1 may be taken as accurate to the first decimal, and will be used as the basis of all calculations.

Obviously, it is not impossible that ytterbium might be decomposed by hitherto unknown or untried methods. However, I have not been able to discover any indication that this is the case, and at present no reason for doubting the elementary nature of ytterbium appears to exist.

Only one oxidation product, the sesqui-oxide, is known. An attempt to obtain lower oxides was unsuccessful. The oxalate was moderately heated in an uncovered crucible until the weight became constant, the residue weighed, and then again ignited in the open crucible before the blowpipe, without any alteration in weight taking place. The oxide thus obtained was identical with the substance prepared by igniting the nitrate.

Before I pass to the description of the ytterbium compounds, I must mention that for the preparation of some of these an earth was used which had an atomic weight somewhat lower than the theoretical.

It was not possible, without sacrificing too much of the material, always to reach the right value. Hence in some analyses the percentage of ytterbium was found to be a little too low. In other cases, on precipitation with oxalic acid, a similar result was obtained, owing to the fact that the oxalate is slightly soluble. As will be proved later, the salt is among the most soluble of the oxalates of the rare earths. Precipitation with ammonia is therefore much to be preferred for analytical purposes.

All determinations of specific gravity were made according to O. Pettersson's directions (*Ber.*, ix., 1859), by weighing in benzene from which the air has been expelled by boiling under diminished pressure. The values given are referred to the temperature of the room. The specific gravity of the benzene was determined independently each time.

(To be continued).

ON RADIUM AND RADIO-ACTIVE SUBSTANCES.

By F. GIESEL.

IN a recent communication to No. 24 of the third annual volume of the *Physikalische Zeitschrift* I have shown that radium has a characteristic pure carmine-red flame colouration and a bright flame spectrum. This latter differs conspicuously from Demarçay's spark spectrum, in that two broad intense lines appear in the orange-red, while the spark-spectrum only exhibits two very faint lines in this region; these lines have not been further investigated. Professor Runge is going to be kind enough to determine the spark-spectrum of radium.

Furthermore, using the pure radium bromide, I had been able to confirm my earlier observation of the constant liberation of bromine (F. Giesel, *V. d. Phys. Ges.*, II. Sitzung, vom January 5, 1900) from the salt containing barium (in addition to the ozonisation of the air). I have now also proved the effect of this liberation of bromine, namely, the formation of radium hydroxide (with alkaline reaction) and radium carbonate formed from it (by the action of the carbon dioxide of the air). The proof that the reaction of the radium bromide becomes alkaline is rendered more difficult because the solution bleaches litmus in consequence of the formation of a salt of hypobromous acid. As this gradual splitting up of the radium bromide proceeds a gas is disengaged. The crystals occlude a part of this gas, and give it up with decrepitation on solution in water. Hence, when solution begins they become milky. Exactly the same processes occur not only in the solid salt but also in the solution. This becomes yellow owing to the presence of free bromine which remains dissolved, and a colourless gas is constantly disengaged. This gas may be collected in the pure state by filling pipettes with the solution of the radium salt and allowing the gas as it escapes to displace the liquid. After Curie's researches on induced activity, it is not surprising to find that this gas is exceedingly active, causes phosphorescence, and in a very short time colours the glass pipettes.

It was most probable that the gas was hydrogen, but a mixture with one-third volume of oxygen in a pipette did not explode when a flame was applied. Should the spectral analysis of the gas, which is now in course of progression, show that the gas does not originate from the decomposition of the water, but that it is a new gas, it must be derived from the radium, and it is to be assumed that it stands in a causative relation to the induction effect of the radium. Thus an insight would be given into the internal processes of the radium atom.

The preparation of pure radium from the raw material is carried out by the method I employed three years ago (*Wied. Ann.*, 1899, lxi, 92; cf. also Ahrens, *Sammlung Chem. und Chem. Tech. Vortr.*, Bd. vii., S. 7 and 8). The raw material consists principally of barium, strontium, and calcium, and only contains very small quantities of radium (in the most favourable case only 0.3 per thousand). After converting into the bromides one crystallisation is sufficient to remove strontium and calcium. After about eight more crystallisations of the mixture of barium and radium bromide from water, the separation of the more easily soluble barium bromide is generally complete. The colouration of the Bunsen flame conveniently indicates the progress of the purification. Fractional precipitation by ammonium carbonate may also be employed for the same purpose. Radium carbonate is precipitated last.

Of other salts of radium, except the sulphate, the only coloured salt which has hitherto been prepared on account of its optical properties is the double cyanide of platinum. It scarcely differs from the salt containing barium which has previously been obtained (*Wied. Ann.*, 1899, lxi, 69; *Verh. d. d. Phys. Ges.*, II. Sitzung, v. Jan. 5, 1900), but even in the liquid becomes strongly dichroic in a few minutes (brown-red to black in crossed crystals).

The pure anhydrous radium bromide exhibits a splendid bluish phosphorescence at first, and has a continuous

spectrum. The phosphorescence decreases after one day in consequence of the appearance of a colouration; but it may be temporarily revived (decolouration produced by heating), but afterwards disappears more and more. Hence, for the demonstration of the strong phosphorescence of the bromide, a preparation containing barium is to be preferred. The Becquerel radiation of the pure radium salts is very considerable, and it is only necessary to mention that previously fused alkali haloids are coloured by them in a few minutes.

With regard to Marckwald's work on polonium (*Berichte*, 1902, xxxv., 2285), I should like to point out that the Curies call special attention to the fact that their polonium sends out chiefly easily absorbed rays, which are partly intercepted even by the thinnest aluminium foil. Marckwald has not investigated the question whether this radiation, which has been thoroughly studied by the Curies, behaves differently from that of his preparation. The polonium prepared by me—besides the easily absorbed rays which are also present (*Wied. Ann.*, 1899, lxi, 93)—at first gives rise to such powerfully penetrating rays, that I could discover in them the magnetic power of deflection of the Becquerel rays (*Wied. Ann.*, 1899, lxi, 834). The polonium gradually loses this radiation, which is rendered perceptible by the ordinary screen of barium-platinum cyanide, but it retains a residual effect, to which attention has already been called (*Berichte*, 1901, xxiv., 3775). Thus, as regards its initial effect, my polonium has nothing to do with Marckwald's body, and hence cannot be considered with it. According to this, Marckwald's observations at present do not contradict earlier information on this point. Also, it seems to me that the assumption that the chief constituent of my polonium is bismuth rendered inactive by induction is not weakened. As before, I can obtain polonium with the properties in question out of a material which has for a long time settled in layers, and, for preference, from the active barium and lead precipitates. The dissipating induced activity is again restored, by continued induction, to bismuth which is embedded in active material.

The residual effect which has been mentioned can be derived from Marckwald's body. This, in any case, may be separated from my polonium, even if it has become almost inactive, in even larger quantity than from the rest of the bismuth often contained in considerable amount in the uranium ores. For the initial activity (induced) of my polonium this body cannot be made responsible, because this activity could not disappear in its presence.

The properties mentioned by Marckwald as characteristic of his body I can confirm, and add that the radiation—as in the case of radium—colours paper yellow in the course of several weeks. Only indications of the phosphorescence of the barium-platinum cyanide screen are to be observed on applying the preparation on the side of the stratum. On the other hand, a screen of Sidot's hexagonal blende (zinc sulphide), prepared with gelatin, reacts in such a way that it is affected as by radium salts, and afterwards emits light.* Even at some centimetres' distance the phosphorescence is easily visible; at a greater distance probably the layer of air acts as an absorbent.

For such easily absorbed rays the screen of zinc sulphide is a valuable means of observation and demonstration. In this way, in some preparations obtained from radium impurities which only possessed relatively small rectilinear radiation, I have been able to demonstrate Rutherford's emanation in a most convincing manner.

If a filter with some centigrams of the substance is laid on the screen, the emanation spreads over a larger extent of the screen and produces a phosphorescence which is driven to and fro by the lightest current of air. By blowing, the direction can be altered at will. If the preparation is placed in a tube, by blowing with the mouth a current of air can be obtained which will act strongly on

* I notice, in addition, that Marckwald (*Phys. Zeit.*, No. 10, IV. Jahrg.) uses zinc oxide as a phosphorescent substance; it is considerably less sensitive.

the screen. The phenomenon cannot be observed with a screen of barium-platinum cyanide or a screen with Balmann's luminous paint. The emanation is not affected by magnetic forces; but, on the other hand, it is affected by electric forces. If the screen is given a negative charge by an influence machine, on bringing the substance wrapped in filter-paper close to it, it becomes luminous, and not uniformly, but the light is concentrated in a peculiar manner into points or rings (screen effect); the luminosity ceases when the preparation is removed, or when the screen is discharged or given a positive charge.

The substance giving rise to the emanation will now be examined. It appears to possess a peculiar chemical nature, as no other of the known strongly active substances or thorium earths reveals an emanation in this manner. The body is found in the ammonia precipitate after separation of the rare earths from pitchblende with oxalic acid. Possibly it is in some way connected with Rutherford's hypothetical body "ThX." The emanation does not appear to be a gas; at any rate, no disengagement of gas from the substance was observed under water.

I obtain the earths which belong essentially to the cerium group (*Berichte*, 1900, xxxiii., 3570; 1901, xxiv., 3776) from other material of constant activity, and it is possible to purify by fractional crystallisation of the oxalates from dilute nitric acid, so that there seems to be some prospect of being able to investigate Debierne's actinium. But thorium is only present to a very small extent, and methods aiming at the separation of it no longer, as before, lead to an increased—but, on the contrary, to a diminished—activity.

Unfortunately, from the radium mother-liquor I can no longer obtain my strongly and constantly active substance (*Berichte*, 1902, xxv., 102), which adheres to the lead, or I can only get traces of it; on the other hand, the emanation body is present here also.—*Berichte*, 1902, xxv., 3608.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING OCTOBER 31ST, 1902.

By SIR WILLIAM CROOKES, F.R.S.,

and

PROFESSOR DEWAR, F.R.S.

To CHARLES FERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolitan Water Act, 1871.

London, November 10th, 1902.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 208 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from O&A. 1st to O&A. 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 208 samples examined by us chemically during the month, all were found to be clear, bright, and well filtered.

There is no indication yet of any break-up of the prolonged drought. The rainfall at Oxford during October was 1.55 inches; the average fall for the month is 2.76 inches, leaving a deficit of 1.21 inches; this, added to the

previous one of 6.63 inches, gives a total deficit of 7.84 inches, or 37.6 per cent on the thirty-five years' average.

Our bacteriological examinations of 536 samples have given the results recorded in the following table; besides these we have examined 38 other samples, from special standpipes, wells, &c., making a total of 574 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 26 samples) ..	314
New River, filtered (mean of 127 samples) ..	11
Thames, unfiltered (mean of 26 samples) ..	8243
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 295 samples) ..	20
Ditto ditto highest	265
Ditto ditto lowest	0
River Lea, unfiltered (mean of 26 samples) ..	243
River Lea, from the East London Company's clear-water wells (mean of 36 samples) ..	17

Of the 458 daily samples taken from the filter-wells of the Metropolitan Water Companies, and examined bacteriologically by us, twenty-seven samples, or 5.8 per cent, were sterile. Four samples contained more than 150 microbes, while twelve samples, or 2.6 per cent, contained more than 100 microbes per c.c. The mean number of microbes in the twelve excess samples was 145, against a corresponding mean of 128 in eighteen samples during September.

Considering the time of year, these figures show that the condition of the London waters is highly satisfactory from a bacteriological point of view; and the same remark also applies to its chemical characteristics.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.

JAMES DEWAR.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

THE following are abstracts of papers received during the vacation, and published, or passed for publication, in the *Transactions*:—

127. "On the Solvent Properties of Mixed Liquids in Relation to the Chemical Characters and Solvent Properties of their Components." By H. M. DAWSON.

The solvent power of mixtures of liquids in its relationship to that of the components has been investigated under conditions such that complicating secondary effects of electrolytic dissociation, high concentration of the solutions, and interaction of the component liquids are avoided.

Measurements of the solubility of iodine were obtained by determining the ratio of distribution between water as standard substance on the one hand, and mixtures of organic solvents on the other. A method of determining this partition ratio is described in which an aqueous solution of potassium iodide is employed instead of pure water. If the iodine concentration in the aqueous solution remains the same in all experiments, the conditions of experimentation correspond almost exactly with the determination of the solubility of a gas in different liquids, the pressure of the gas being kept constant. The organic solvents chosen were such as would not be expected to have any action on each other, or on the dissolved iodine. In these circumstances, it is found that in the majority of cases the solvent power of the mixture is less than the sum of the solvent powers of the components; sometimes, however, it is equal to, or greater than, the sum of the solvent powers of the components. No case of a maximum or minimum solvent power, such as that observed with

alcohol-water mixtures in regard to hydrogen, oxygen, carbon monoxide, and carbon dioxide has been found.

128. "Notes on Luteolin and Apigenin." By A. G. PERKIN.

Kiliani and Mayer (*Ber.*, 1901, xxxiv., 3577) have recently asserted that the author's tetrabenzoyl-luteolin (*Trans.*, 1896, lxix., 209) is in reality a tribenzoyl derivative. A re-examination of the original preparation shows that the statement of Kiliani and Mayer is incorrect, and that the substance is without doubt tetrabenzoyl-luteolin. Further experiments with reference to the tinctorial properties of apigenin and chrysin confirm the previous results of the author (*Trans.*, 1897, lxxi., 806).

129. "Note on a Simple Form of Landsberger's Apparatus." By E. B. LUDLAM.

A description is given of an apparatus which has proved most satisfactory to work with. The tube in which the rise of temperature is measured is graduated and placed in the neck of the flask containing the boiling solvent, the vapour of which enters it from below by means of a simple valve which serves the double purpose of regulating and breaking up the stream of vapour, and also of preventing the solution from running back into an intermediate tube, placed between the incrometer and the flask, when the latter is cooled.

The graduated tube is also provided with an annular trough which serves as a trap and catches the liquid condensed in the upper part of the tube, conveying it to the condenser, thus making the increase of volume in the tube much slower than it otherwise would be.

130. "The Action of Substituting Agents on Benzeneazo- β -naphthol." By J. T. HEWITT and S. J. M. AULD.

Bromine added to benzeneazo- β -naphthol suspended in glacial acetic acid furnishes β -bromobenzeneazo- β -naphthol, a result also obtained by Margary (*Gazzetta*, 1883, xlii., 438), who assigned to the substance the melting-point $160-161^\circ$, but which the authors, like Bamberger, find to be $172-173^\circ$. In presence of sodium acetate, such a high temperature is necessary before the bromine will attack the azo-compound that the results obtained lead to no definite conclusion as to the structure of benzeneazo- β -naphthol.

Nitric acid in presence of concentrated sulphuric acid nitrates the azonaphthol in the benzene nucleus (para-position), dilute nitric acid attacks benzeneazo- β -naphthol on warming, giving 1:6 dinitro- β -naphthol as the chief product, and β -nitrobenzeneazo- β -naphthol in smaller amount.

With strong acids, benzeneazo- β -naphthol behaves as a quinone-hydrazone; with dilute acids (nitric) or weak acids (acetic) its reactions are more those of an azo-naphthol.

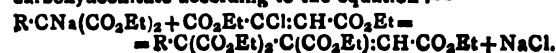
131. "The Condensation of Dimethylaminobenzaldehyde with β -Naphthol." By J. T. HEWITT, A. J. TURNER, B.Sc., and S. W. BRADLEY.

When hydrochloric acid is added to a solution of dimethylaminobenzaldehyde and β -naphthol in cold glacial acetic acid, crystals of the composition $\text{HCl} \cdot (\text{CH}_3)_2\text{N} \cdot \text{C}_6\text{H}_4 \cdot \text{CH}(\text{C}_2\text{H}_5\text{OH})_2$ are deposited; these darken at about 150° and melt at 215° with decomposition. If, however, a mineral acid be added to a hot glacial acetic acid solution of the aldehyde and β -naphthol, a salt of the corresponding anhydride is produced.

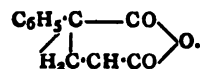
Attempts at oxidation were without result; even bromine does not yield an oxonium salt, but gives the bromide of a bromo-substitution product, $\text{C}_{20}\text{H}_{22}\text{BrON} \cdot \text{HBr}$ (m. p. 196° , uncorr.).

132. "The Action of Ethyl Chlorofumarate on Mono-alkylmalonic Esters." By S. RUHEMANN.

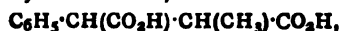
The author finds that, in the presence of sodium ethoxide, ethyl chlorofumarate reacts with the mono-alkyl derivatives of ethyl malonate to yield homologues of ethyl carboxyaconitate according to the equation:—



The quantity of the unsaturated ester which is produced is small, and decreases as the radicle in the substituted malonic ester increases. Of several homologues prepared, the author has studied especially ethyl phenyl-carboxyaconitate, $\text{C}_6\text{H}_5 \cdot \text{C}(\text{CO}_2\text{Et})_2 \cdot \text{C}(\text{CO}_2\text{Et}) : \text{CH} \cdot \text{CO}_2\text{Et}$, and finds that, on hydrolysis, it loses two molecules of water, and yields a compound, $\text{C}_{17}\text{H}_{15}\text{O}_5$, which is the anhydride of a dicarboxylic acid. This anhydride is not an unsaturated compound, but a derivative of trimethylene having the formula—



On reduction with sodium amalgam, this compound yields α -phenylmethylsuccinic acid,—



(m. p. 193°), which is identical with the acid described by Zelinsky and Buchstab (*Ber.*, 1891, xxiv., 1876).

133. "Di-sec-octyl Tartrate and Di-sec-octyl Dibenzoyl Tartrate." By J. MCCRAE.

Di-sec-octyl tartrate was prepared from ethyl octyl tartrate and octyl alcohol by the hydrochloric acid method. It is an oil which boils at 225° (20 m.m.), and has $[\alpha]_D^{18} = 7.06^\circ$. The dibenzoyl derivative was prepared by adding on the ester with excess of benzoyl chloride, and was purified by precipitation with water from an alcoholic solution. It is an oil with $[\alpha]_D^{25} = -43.94^\circ$. The rotations of these two compounds are compared with those of other tartaric esters.

134. "Glycogen from Yeast." By A. HARDEN and W. J. YOUNG.

The glycogen of yeast was extracted by grinding the yeast with sand, pouring the mass into boiling water, and purifying the glycogen by a combination of the methods of Clautriau and Pfüger. Expressed yeast juice was employed. The glycogen, free from ash and nitrogen, was compared with glycogen extracted by Pfüger's method from rabbit liver and from oysters.

All three samples, dried at 100° in a vacuum over phosphorus pentoxide, were found to have the composition $\text{C}_6\text{H}_{10}\text{O}_5$ and not $6\text{C}_6\text{H}_{10}\text{O}_5 + \text{H}_2\text{O}$. Glycogen dried at 100° in air always retained a certain amount of water.

The specific rotations were found to be, rabbit glycogen, $[\alpha]_D^{15} = +191.1^\circ$; oyster glycogen, $[\alpha]_D^{15} = +191.2^\circ$; yeast glycogen, $[\alpha]_D^{16.5} = +198.3^\circ$.

In view of the low concentrations of the solutions rendered necessary by their opalescence, this difference cannot be considered as characteristic.

The glycogen from yeast appears to be identical with animal glycogen in composition and optical activity. Slight differences appear to exist in its behaviour towards dilute acids, in the reaction with iodine, and in the opalescence of the aqueous solutions, but these are also exhibited by glycogens derived from various animal sources.

135. "The Action of Acetylene on the Acetates of Mercury." By E. BURKARD, Ph.D., and M. W. TRAVERS, D.Sc.

The authors have investigated the compounds produced by the action of acetylene on mercurous and mercuric acetates respectively, which were briefly described by the late Dr. R. T. Plimpton (*Proc.*, 1892, viii., 109).

The mercurous compound, which is explosive, has the formula $\text{C}_2\text{Hg}_3\text{Hg}_2\text{O}$, and resembles the already well-known acetylides of silver, copper, and mercury.

The mercuric compound appears to be a definite basic acetylide of the formula $3\text{C}_2\text{Hg}_2\text{HgO} \cdot 2\text{H}_2\text{O}$ or $3\text{C}_2\text{Hg}_2\text{Hg}(\text{OH})_2$; it is non-explosive.

Both compounds yield acetylene when treated with acids, and a mixture of di-iodoacetylene and tetraiodoethylene when treated with iodine. Hence it appears that the oxygen and hydrogen in the compounds are connected with the mercury rather than with the carbon.

136. "The Decomposition of Water Vapour by the Electric Spark." By D. L. CHAPMAN and F. A. LIDBURY.

The experiments described show that when electric sparks are passed through water vapour the decomposition which takes place is considerable. The products of decomposition are partly separated from one another, and take up different positions in the tube. The separation which occurs cannot be entirely accounted for by electrolysis for the following reasons:—

1. Hydrogen collects at both electrodes and oxygen in the middle of the spark gap.

2. The hydrogen which collects at the cathode is very largely in excess of that required by Faraday's law.

137. "The Chlorination of the Dichlorotoluenes in presence of the Aluminium-mercury Couple. The Constitution of the Trichlorotoluenes." By J. B. COHEN and H. D. DAKIN.

The six dichlorotoluenes were chlorinated, using the aluminium-mercury couple as carrier. In order to identify the products formed, the six trichlorotoluenes were prepared synthetically, together with their nitro- and dinitro-derivatives and the trichlorobenzoic acids which they yield on oxidation. The results show that the six dichlorotoluenes, on chlorination, give five out of the six possible trichlorotoluenes.

138. "The Constitution of the Nitro- and Dinitro-derivatives of the Dichlorotoluenes." By J. B. COHEN and H. D. DAKIN.

The authors have determined the constitution of the nitro- and dinitro-derivatives of the six dichlorotoluenes which they had previously prepared (*Trans.*, 1901, lxxix., 1111). They find that in the case of the mono-substitution derivatives, the chlorine atoms possess a greater influence than the methyl group in determining the position of the entering nitro-group, whilst in the case of the dinitro-compounds the position of the first entering nitro-group appears to be of primary importance in determining the position to be occupied by the second nitro-group, for in all the six dichlorodinitrotoluenes the nitro-groups are in the *meta*-position to one another.

139. "Iodonium Compounds of the Type IR'R''R''', and the Configuration of the Iodine Atom." By H. PETERS.

Further investigation of the phenyl-*p*-tolylidonium bromocamphorsulphonate described in a previous note (*Proc.*, 1900, xvi., 62) has confirmed the conclusion that it cannot be resolved into salts of enantiomorphously related bases, from which the inference may be drawn that the three iodine valencies are situated in one plane, two of them being symmetrically situated with respect to the third.

In the preparation of phenyl-*p*-tolylidonium iodide from iodoso-*p*-toluene and iodoxybenzene small quantities of di-*p*-tolylidonium iodide are formed owing to secondary reactions; similarly, the crude phenyl-*p*-tolylidonium iodide, prepared from iodosobenzene and iodoxy-*p*-toluene, contains small quantities of diphenylidonium iodide. The formation of these subsidiary products is not due to extra-molecular change of the radicals of the phenyl-*p*-tolyl salt, and it is shown that the latter is really the salt of a mixed idonium base.

Phenyl-*p*-tolylidonium nitrate, $\text{Ph}(\text{C}_6\text{H}_4\cdot\text{Me})\text{I}\cdot\text{NO}_3$, crystallises in prisms melting at 117°. The chloride, $\text{Ph}(\text{C}_6\text{H}_4\cdot\text{Me})\text{I}\cdot\text{Cl}$, crystallises in needles, and melts at about 193°.

Di-*p*-tolylidonium iodide, $(\text{C}_6\text{H}_4\cdot\text{Me})_2\text{I}$, forms well-defined, octahedral crystals and decomposes at 143–153°.

Di-*p*-tolylidonium bromocamphorsulphonate, $(\text{C}_6\text{H}_4\cdot\text{Me})_2\text{I}\cdot\text{C}_{10}\text{H}_{14}\text{BrO}_4\text{S}$, crystallises in needles melting at 185–186°; it is sometimes obtained in hydrated dodecahedra, very like the crystals of the corresponding salt of the mixed base.

Diphenylidonium bromocamphorsulphonate,—
 $(\text{C}_6\text{H}_5)_2\text{I}\cdot\text{C}_{10}\text{H}_{14}\text{BrO}_4\text{S}$,

forms hydrated dodecahedra, the anhydrous salt melting at 165–168°.

140. "Influence of Substitution on the Formation of Diazoamines and Aminoazo-compounds." Part II. By G. T. MORGAN, D.Sc.

Mixed diazoamines containing a naphthalene nucleus are readily obtained from the diazotised *o*- and *m*-nitranilines and 1-chloro- β -naphthylamine. The properties and reactions of several are described in detail in the paper.

141. "Observations on the Phenomena and Products of the Decomposition of Normal Cupric Acetate when Heated." By A. ANGEL and A. V. HARCOURT.

The authors give a summary of the numerous researches on this subject. One phenomenon which had not been noted previously is the formation of a bright coating of copper in the test-tube in which the salt $[\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2\cdot\text{H}_2\text{O}]$, is heated. As this salt does not fuse this must be due to a gaseous compound of copper, proved by the authors to be cuprous acetate, which volatilises at about 200°, and forms, especially when sublimed in hydrogen, feathery white crystals.

The salt was heated generally in a tube in an oil-bath, and kept nearly vacuum by a Sprengel pump, and the gases collected. These were found to consist wholly of the oxides of carbon in approximately the ratio $4\text{CO}_2:\text{CO}$. The liquid products consisted of water and acetic acid with a trace of acetone. The fixed residue is of a paler or deeper red-brown colour, and consists of metallic copper mixed with a bulky black substance, the composition of which may be represented by the formula $\text{Cu}_2\text{H}_4\text{O}_4$.

142. "The Resolution of β -Hydroxybutyric Acid into its Optically Active Components." By A. MCKENZIE.

In a preliminary note (*Proc.*, 1901, xvii., 213), the author mentioned that he had obtained *l*- β -hydroxybutyric acid from the inactive acid by aid of the quinine salt.

The *d*-acid has since been isolated by means of the strychnine salt, the solvent being ethyl acetate. It was found that for it $[\alpha]_D^{20} = +24.3^\circ$ ($c = 2.226$).

Change of concentration has little influence on the value for the specific rotation of the acid (Magnus-Levy) and the salts are active in the same sign as that of the acid but to a less degree. In these respects, β -hydroxybutyric acid differs from most other aliphatic oxy-acids.

When the *l*-acid is heated at 100°, it is partly converted into an anhydride.

When the inactive potassium and sodium salts were respectively injected subcutaneously into dogs, the author found from examination of the urine (1) that acetoacetic acid and acetone were present, (2) that a small portion of the β -hydroxybutyrate was unattacked, and (3) that this product was laevorotatory.

Partial resolution of the acid was also obtained by esterification with *l*-menthol and *l*-borneol.

143. "The Rate of Decomposition of Diazo-compounds. Part I. Diazo-compounds of the Benzene Series." By J. C. CAIN and F. NICOLL.

The decomposition of an aqueous solution of a diazo-salt is in most cases a simple monomolecular one, conforming to the expression—

$$C = \frac{1}{t} \log \frac{A}{A-x}$$

The authors have measured the rate of decomposition of diazo-salts from various bases, and find that at temperatures between 20° and 60° the diazo-salts from aniline and the toluidines conform to the above law, thus confirming some results of Hantzsch at 25° (*Ber.*, 1900, xxxiii., 2517), but being quite opposed to the work of Hauser and Muller at 40° and 64° (*Bull. Soc. Chim.*, 1892, [iii.], vii., 721; 1893, ix., 353).

The authors maintain that the assumption by the latter chemists of a specific "retarding action" of the phenol formed cannot be upheld.

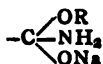
A table of constants of the diazo-salts from eight amines shows the very great stability of the diazo-salts from substances containing an acid group (NO_2 or SO_3H).

The diazo-salt from *p*-amidoacetanilide follows the law only in acetic acid solution, as in presence of mineral acid the acetyl group is saponified.

Of the tetrazo-salts of the diamines, only that from dichlorobenzidine follows the law. The others give in the case of the tetrazo-salts from benzidine and tolidine diminishing, and in the case of that from dianisidine, increasing, values for C_{∞} , pointing to the probability of the formation of an intermediate substance containing still a diazo-group.

144. "Studies upon the Action of Sodamide and Acetyl Substituted Sodamides upon Organic Esters." By A. W. TITHERLEY, D.Sc., Ph.D.

Sodamide appears to form addition compounds containing the grouping—



when allowed to act on esters in benzene solution if the temperature is kept low. On warming, further action at once takes place, which varies according to the nature of the ester. With esters of benzoic and other purely aromatic acids, an alcohol splits off and a substituted sodamide results.

With esters of fatty and phenyl aliphatic acids, the final result of the reaction is a condensation effected on very similar lines to those brought about by sodium ethoxide, and probably by a similar mechanism.

Phenyl benzoate behaves quite differently from the ethyl ester in giving sodium dibenzamide and phenoxide, and the reaction proceeds with great readiness. Ethyl oxalate gives an intermediate product which with water yields sodium oxamate.

The action of sodamide with ketones is very different in the aliphatic series when condensations are brought about from that in the pure aromatic, where there is no action. Acetone with sodamide gives, in presence of benzene after a vigorous reaction, a jelly consisting essentially of the sodium derivatives of the enolic forms of mesityl oxide, phorone, and isophorone.

The interaction of acyl-substituted sodamides and esters depends largely on the nature of both these substances, which, if aliphatic, induce various complex changes. In the pure aromatic series, a simple definite change occurs which conforms approximately to the reversible system $R \cdot CO \cdot NHNa + R' \cdot CO_2R' \rightleftharpoons (R \cdot CO)_2N \cdot Na + R' \cdot OH$, but in all probability the mechanism of the reverse reaction is more complex.

The reaction is capable of modification in numerous ways, and the acyl-substituted sodamide may be prepared from the amide and alcoholic sodium ethoxide, or (best) from sodamide. A very good yield of dibenzamide is obtainable from sodium benzamide and ethyl benzoate.

NOTICES OF BOOKS.

Directions for Laboratory Work in Physiological Chemistry; for the Use of Students in the University and Bellevue Hospital Medical College. By HOLMES C. JACKSON. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1902. Pp. vi.-62. 8vo.

THIS well-devised little volume contains the usual tests and simple experiments made in giving instructions to classes on the following subjects:—Carbohydrates, fats, proteids (general, simple, and compound), albumenoids, muscular tissue, bone, digestion (salivary, gastric, and pancreatic), bile, blood, milk, and urine in all its aspects.

The directions for experimental work are terse and lucid; they assume a general knowledge of the subject, and the method of treatment necessitates consultation of some standard text-book of physiological chemistry. Neither on the title-page nor in the modest preface is any particular book named, but occasional reference is made in the text to the work by Olof Hammarsten, translated

from the German by John A. Mandel, a third edition of which was issued in 1899 by the publishers of the work under review. Dr. Mandel is Professor of Physiological Chemistry in the same institution as the author of this book. Although prepared for the special service named, the work will be found helpful elsewhere.

Occasional interrogation points (within parentheses) inserted in the text direct the attention of students using the volume to consideration of the significance of a reaction or the nature of a product; this simple expedient replaces the questions which sometimes disfigure the pages of text-books, and which frequently seem inane.

The customary designation of special tests by the names of those first announcing them leads to possible difficulty in their application, but the author has avoided this by briefly describing each test. In most cases simplification of language results, but in others this is questionable; the use of glacial acetic acid and concentrated sulphuric acid in examining albumin solutions is hardly simplified by calling the method "Adamkiewicz test," though it is briefer.

The book is well printed, but lacks an index; it is interleaved with blank pages for notes.

Lead Smelting: The Construction, Equipment, and Operation of Lead Blast-furnaces, and Observations on the Influence of Metallic Elements on Slags, and the Scientific Handling of Smoke. By MALVERN WELLS ILES. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1902. Pp. viii.-228. 12mo. Illustrated.

DR. ILES condenses in this volume the results of twenty years' practical acquaintance and successful experience in the smelting of lead; he remarks that most of the literature that precedes him emanates from students of chemistry and metallurgy well versed in the theories of these sciences, but who have not enjoyed the advantage of personal contact with the practical problems confronting lead smelters. Dr. Iles does not undervalue the importance of a profound knowledge of theories, but believes that the experienced gained in applying the theories is the prime factor in achieving financial success; he further writes of his book as follows:—

"Without attempt at literary embellishment, and with perhaps censurable disregard for niceties of diction, I go directly to the pith of the subject. My endeavour shall be to give novelty to that which was old, condensation to that which was diffuse, perspicacity to that which was obscure, and accuracy to that which was recondite. I shall attempt truthfully to relate what *has been* and *is*."

This undertaking is fully carried out with a wealth of detail based on experience that makes the volume a rare one. The author devotes the first hundred pages to lead blast-furnaces, their construction, working, and supervision, as well as the method of calculating charges, making daily reports, and checking errors.

In a valuable chapter the "metallurgical results" are discussed; the importance of a "bag-house" is shown, as well as of "slag-settlers," whereby 70 per cent of the silver previously lost in the slag is saved.

Every chapter abounds in records of actual experiments, many pages bristle with figures; the scientific handling of smoke, the influence of metallic elements, the use of roasting-furnaces, antimonial lead as found in the western part of the United States; these form separate chapters of this intensely practical volume.

The book is illustrated with drawings of furnaces obviously accurate, and concludes with an uncommonly full index.

H. C. B.

The Earth in Relation to the Preservation and Destruction of Contagia. By GEORGE VIVIAN POORE, M.D. (Lond.), &c. London, New York, and Bombay: Longmans, Green, and Co. 1902. Pp. 257.

THIS volume is made up of the Milroy Lectures delivered by the author at the Royal College of Physicians in 1899, to-

gether with other papers on sanitation, an Address to the Royal Medical and Chirurgical Society, &c., and its chief object appears to be to advocate the direct application of dung to the soil. The enormous waste of nitrogen which is continuously going on in all our great cities, owing to our modern methods of sanitation, by which sewage is discharged into the sea and lost, is well known, and has been the subject of much discussion and serious thought. Dr. Poore urges the direct application of such matter to the soil itself, and he brings forward many arguments in its favour, backed up by practical instances of its efficacy.

There is a great deal to be said on both sides; there can be no doubt that owing to the drying action of the wind and sun, many pathogenic microbes are widely disseminated in the form of dust; on the other hand, it is equally certain that enormous numbers of these bacteria are killed by the action of these same agencies.

In the country, quite away from large towns, we do not dispute that this method of getting rid of sewage matter is a convenient and possibly harmless one, but we cannot agree that such a method is to be advocated in proximity to large centres of population.

The author brings forward a number of instances in which this means of sewage disposal has been adopted with no untoward results, but has, on the contrary, been found to answer successfully. There are, however, some points to which we must take exception; on several occasions he mentions China as a good example of this, "the dry" method of sewage disposal, and states that physically and economically they (the Chinese) are "going strong," and are likely to continue; surely it is too much to ask us to take the Chinese as a standard of good sanitation; we are not aware of the actual death-rate in China, but from the periodical accounts we receive of deadly epidemics by which even millions of persons are swept off, we cannot accept this example as a proof of the efficacy of the system from the sanitary point of view.

It may be that if the sewage had not been utilised in this manner the inhabitants would have died of starvation, or that the population would not have become so dense; but this is quite another question.

Another point to which we must draw attention is Dr. Poore's description of his garden and the well in the middle of it. This garden is a little over 1 acre in extent, and is manured with the daily faecal matter and house refuse from 100 persons. We quite believe that the practical gardening results are excellent; but in the middle of this garden there is a shallow well, the bottom of which is only five and a half feet from the surface of the ground, and though from its construction no water can enter the well except through the bottom, the author drinks the water without any hesitation or misgiving, because he knows that there are no leaking sewers or cesspools in the immediate neighbourhood! Why, the whole garden is little better than a cesspool! The author admits that his well has retrogressed in bacterial purity, but maintains that this retrogression is caused by insects, such as wood-lice and spiders, which he found under the cover of the well. This is certainly a case of "straining at a gnat and swallowing a camel."

The author then asks, "I wonder if bacteria of the coli group are found in wood-lice, spiders, and water-fleas?" "I wonder what is the life history of a water-flea?" Surely the answers to such simple questions as these could have been ascertained without difficulty.

However, apart from a rather pronounced leaning towards the "dry method" and earth-closets, there is much in this volume of interest and value. The connection between various diseases and the soil as a possible means of contagion has been thoroughly examined.

With regard to contagion from milk, the author does not bind himself to any definite opinion, but he is inclined to think that city children will be improved on the whole by giving them as much new milk as they can take.

The Cyanide Process for the Extraction of Gold; and its Practical Application on the Witwatersrand Goldfields and Elsewhere. By M. ESSLER. Third Edition. Revised and Enlarged. London: Crosby Lockwood and Son. 1902. Pp. 184.

The result of the additions due to the extension of, and improvements made in, the cyanide process is an increase of about forty pages of new matter in this volume, with some additional illustrations.

We are glad to see that the demand for books on this subject is increasing; there is no doubt that the cyanide process has been the saving of many a mine. It is only free gold that is caught on the plates, and even then it must not be in too finely divided a state; but with the advent of this process practically all the gold in the ore can be recovered, leaving only a few grains in the tailings; in fact, many heaps of tailings which were looked upon as of no worth whatever have become, since the introduction of cyaniding, a very valuable and important asset.

As the successive editions of this book are following so rapidly, we need not add much to our remarks made on the former editions; the value of this work is shown by its success. An important addition, however, has been made in the chapter on "The Treatment of Sulpho-Telluride Ores"; this class of ore occurs in considerable quantities in some of the Australian goldfields, and many mines depended for their very existence on the discovery of a practical method for the extraction of the gold.

In the Kalgoorlie district of Western Australia the Diehl process seems to be the most prominent; after passing under the stamps, the pulp is run over concentrators, and the tailings are ground to such a fineness that they will pass through a sieve carrying 220 holes to the linear inch. After settling and drawing off the excess of water, the pulp is agitated with cyanide of potassium and bromocyanide for twenty-four hours; the gold solution is then separated by filter-presses, and sent to the zinc boxes for reduction. The concentrates are roasted in an automatic furnace, and then ground very fine and treated in the same manner as the bulk of the ore.

The Marriner process consists of dry crushing, roasting, and extraction of the gold by the wet cyanide method; the extraction averages 92½ per cent.

The Riecken process for treating telluride and sulphide ores is an electro-chemical system for the recovery of the precious metals from ores by a single operation without filtration.

The bromo-cyanide process, better known as the Sulman-Teed, involves the chemical reaction—



and the advantage claimed for it over the ordinary cyanide process is its great superiority in "potential" cyanide. The works for treatment by this process do not differ from the ordinary cyanide works. The consumption is 1 lb. of potassium cyanide and 0.33 lb. of cyanogen bromide per ton of ore treated, and the extraction is said to be 90 per cent.

The salient features of another process known as the Cassel-Hinmann are the conversion of the bromine into such a form that it can be shipped as a common salt, requiring only solution in water to make it ready for use; leaching in open vats without creating noxious fumes; the generation of nascent bromine in the interstices of the pulverised ore; the recovery of the bromine, and the ease and completeness of the precipitation of the gold from solution.

The book is of great practical value, and should be in the hands of all gold-mine managers and engineers, while it would not be amiss if directors were to make themselves familiar with its contents.

Institute of Chemistry.—The President's Soirée will take place at the Galleries of the Royal Society of British Artists, Suffolk Street, Pall Mall, on Wednesday, December 10th, at 8.30 p.m.

CORRESPONDENCE.

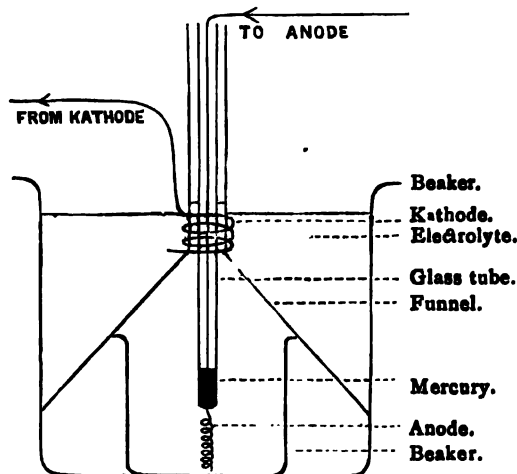
POTASSIUM PERSULPHATE.

To the Editor of the Chemical News.

SIR,—The following is a description of a simple method of quickly preparing some potassium persulphate electrolytically.

The writer used a 400 c.c. beaker. In it he placed a 50 c.c. beaker to receive the deposited persulphate. Over the 50 c.c. beaker was placed a funnel. Through the neck of the funnel was passed a narrow glass tube having a small piece of fine platinum wire fused in to serve as an anode. The anode was connected to the circuit by mercury inside the glass tube.

The cathode consisted of two or three turns of platinum wire round the neck of the funnel.



The electrolyte consisted of saturated potassium sulphate in sulphuric acid of density 1.2.

A current of 1.5 ampères was passed through a lamp resistance.

The salt began to deposit after about ten minutes. The temperature was kept at about 14° C.

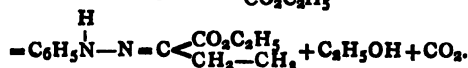
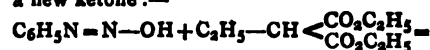
Dr. Karl Elbs gives a good description of persulphate preparations in his recently published book of Electrochemical Preparations.

The apparatus here described is a modification of a test-tube method which he gives.

"S. C." probably had the solution in the porous pot too dilute, and allowed it to get above 20° C., since he used a current of 4 ampères.—I am, &c.,

L. A. G.

Action of the Substituted Malonic Ethers on the Diazo Chlorides.—M. Favrel.—In the action of the methylmalonate of ethyl on the diazo chlorides, the methyl group remains fixed to the central carbon, and at the same time there is an elimination of CO₂ and of methylic alcohol. Ethylmalonate of ethyl reacting in the same manner on the diazo chlorides gives hydrazones of a new ketone:—



Action of Acetylacetone and of its Derivatives on Substitution on the Diazoic and Tetrazoic Chlorides.—M. Favrel.—The author describes a number of bodies prepared by the above means, and gives their analyses and formulae.—*Bull. Soc. Chim.*, Series 3, xxvii., No. 8.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

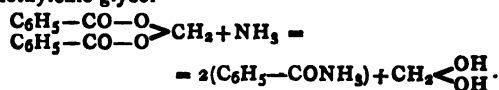
NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxv., No. 17, October 27, 1902.

Synthesis of the Anhydrous Hydrosulphites of the Alkalis and the Alkaline Earths.—Henri Moissan.—Sulphurous anhydride at ordinary temperatures and under certain conditions of pressure reacts on the hydrides of the alkali and alkaline earthy metals in such a manner as to form anhydrous hydrosulphites. This synthesis takes place with an evolution of hydrogen and according to the following equation:— $2\text{KH} + 2\text{SO}_2 = \text{K}_2\text{S}_2\text{O}_4 + \text{H}_2$. All the hydrosulphites are soluble in water and possess energetic reducing properties, identical with those which Schutzenberger noticed for the hydrated sodium hydrosulphite. In all cases they are produced by direct union of sulphurous anhydride and the metal, hydrogen being liberated. The syntheses of these anhydrous compounds verify M. Bernthsen's formula for the hydrated sodium hydrosulphite.

Volumetric Estimation of Tannin and Analysis of Woods and Tannic Extracts.—Albert Thompson.—Tannin when in presence of solutions of caustic alkalis, such as soda or potash, rapidly absorbs oxygen. The author's estimation therefore depends on the following conditions:—(1) Hydrogen peroxide dissociates totally into water and oxygen under the influence of chemically pure lead peroxide and in presence of a concentrated solution of soda or potash. The lead peroxide should be obtained by the treatment of pure minium with nitric acid. (2) The oxygen thus set free is rapidly absorbed by the tannin when the latter is added to the alkaline hydrogen peroxide before the addition of the lead oxide. (3) The tannin being once saturated, the dissociation of the peroxide continues as if the former body were not present, and all the excess oxygen is liberated. (4) 1 grm. of pure anhydrous tannin absorbs 20 c.m. of oxygen at 0° and 760°. (5) Finally, the tannin is soluble in alcohol at 90°, whilst the greater part of the mineral and peptic substances which accompany it are insoluble. The results obtained represent the whole of the tannin which the skin assimilates.

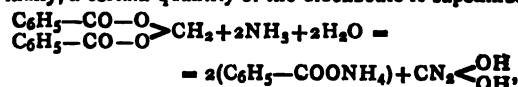
A New Compound of the Group of Hexamethylene-tetramine.—Marcel Descudé.—Methylene-dibenzoate behaves towards ammonia gas in the same way as the ether salts. One portion forms benzamide, the other methylenic glycol—



This unstable glycol at once decomposes into water and formic aldehyde, which complicates the reaction. The formic aldehyde reacts on the excess of ammonia to give hexamethylene-tetramine and a further quantity of water.



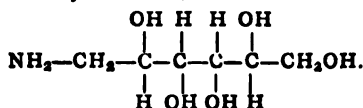
Finally, a certain quantity of the dibenzoate is saponified.



and when the action is complete a mixture of benzamide, ammonium benzoate, and hexamethylene tetramine is present.

A New Base derived from Galactose.—E. Roux.—In a preceding paper the author stated that by reducing sugar oximes he obtained polyalcoholic bases, and one of these, glucamine, he described. The same method applied to the oxime of galactose gives another base, isomeric with the preceding, which is called by analogy galacta-

mine. Its general properties are similar to those of the glucamine already described, and its formula is—



This substance is in the form of a colourless mass of crystalline appearance, very soluble in water, and but slightly soluble in boiling alcohol. It melts at about 139°, and on analysis gives numbers corresponding to the formula $\text{C}_6\text{H}_{13}\text{NO}_5$. Its rotatory power in a 10 per cent solution is -2.77° without multirotation. Its action on metallic salts is similar to that of glucamine, and it does not give a crystalline compound with copper sulphate. It is a strong base which will displace ammonia.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxvii., No. 8.

Aluminates of Barium used as a Disencrustant.—G. Arth.—This product is obtained from sulphate of barium and bauxite. A "frit" is formed, which gives up to water a soluble portion containing alumina and baryta, and leaves an insoluble residue strongly charged with peroxide of iron. The solution is what is used commercially, and the author has made a number of experiments with a view to obtaining some practical hints as to the best manner of using it. He found that the solution as supplied, gives a white deposit, even when kept closely stoppered, and this deposit goes on forming for at least two months, and its strength drops from 5°B. to 4°B. The solution is used principally with waters containing sulphate of lime before entering the boiler, and the reaction is generally admitted to be $\text{BaO} \cdot \text{Al}_2\text{O}_3 + \text{CaO} \cdot \text{SO}_3 = \text{BaO} \cdot \text{SO}_3 + \text{CaO} \cdot \text{Al}_2\text{O}_3$, both compounds formed being equally insoluble. However, this reaction does not agree with what has been observed in practice, viz., that it requires considerably less baryta than is present according to the equation to purify a given water; that is to say, the decomposition is not effected molecule for molecule between the baryta and the sulphuric acid present. The author has observed that no matter what proportions of sulphate of lime and aluminate of baryta are present, after the reaction there is always lime in the precipitate and in the liquid in which the latter is formed. The precipitate is never formed exclusively of sulphate of barium and aluminate of lime. As a result of experiments he found that it is quite possible to precipitate a given quantity of sulphate of lime with a theoretically insufficient amount of aluminate of barium, but the liquid left after the operation is never quite free from soluble compounds.

Derivatives of β -Methylcyclohexanone.—L. Tétray.—The author has obtained a new secondary product of β -methylcyclohexanone by condensation, viz., benzylidene- β -methylcyclohexanone; its composition is $\text{C}_{14}\text{H}_{20}\text{O}_2$. Oxidation of this body by permanganate of potassium gives exclusively benzoic and β -methyladipic acids. Reduction of benzylidene-methylcyclohexanone by sodium amalgam gives benzyl-methylcyclohexanone.

Action of Malonic Ethers on the Diazoic and Tetrazoic Chlorides.—M. Favrel.—The author finds that the action of the tetrazoic chlorides on the malonic ethers is comparable with that of the same ethers on the diazoic chlorides. In both cases there is elimination of water, and the formation of bodies having the composition of the corresponding hydrazones.

Action of the Alcoylacetylacetones on the Diazoic and Tetrazoic Chlorides.—M. Favrel.—These experiments were made with methylacetylacetone and ethylacetylacetone; the bodies obtained are described, and their analyses and formulae given.

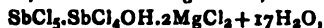
The Detection of the Fatty Acids in Contaminated Waters.—H. Causse.—Already noticed.

New Case of Diastasic Hydrogenation.—M. Pozzi-Escot.—The author has observed that M. de Rey-Pailhade's philothion gives off a notable quantity of sulphuretted hydrogen in the presence of free sulphur; that this philothion gives up hydrogen to selenium and phosphorus; that the action of hydrogenases on oxydases are reciprocal, and that this latter fact explains the destruction of the ceno-oxydase during the fermentation of wine.

Some Lecture Experiments.—F. Bodroux.—An intimate mixture is made of dry magnesium filings with its own weight of pulverised iodine, so as to obtain a homogeneous powder. No reaction takes place; but if a few drops of water are allowed to fall on the mixture, each one causes a violent reaction at the point of contact. Iodide of magnesium is formed, and the disengagement of heat is such that a portion of the iodine is volatilised. The same phenomenon is observed if we use zinc powder with two parts of iodine; but the most brilliant experiment is with aluminium. Six grms. of iodine and 1 grm. of aluminium powder are intimately mixed in a mortar, and a drop of water dropped in; the reaction takes place immediately; a torrent of iodine fumes comes off, the point of contact becomes inflamed, and the whole mixture burns with a yellow flame. The residue consists almost entirely of alumina. The reaction is explained in the following manner:—The drop of water causes the formation of iodide of aluminium at the point of contact. This formation is accompanied by the evolution of sufficient heat to volatilise the iodine formed; now the vapour of this compound is spontaneously inflammable in the air, and produces alumina, setting the iodine free. The reaction spreads, the iodide of aluminium formed is destroyed immediately; all the iodine is volatilised, and the whole of the metal transformed into oxide.

MISCELLANEOUS.

The Double Salts of Pentachloride of Antimony.—R. F. Weinland and Fr. Schlegelmich.—In a similar manner to SnCl_4 , perchloride of antimony easily forms chlorantimonates; however, the formulae are more complicated since the salts formed are rather basic. One molecule of SbCl_5 and half a molecule of the other chloride are dissolved in a sufficient quantity of warm, 15 per cent, HCl ; the yellow liquid formed gives crystals of the double salt when cooled; with magnesium and calcium the cooling must be considerable. The chlorantimonates are of a pale greenish-yellow colour, and are deliquescent with the exception of the ammonium salt; they are decomposed by water, especially when heated, giving antimonous acid. When calcined they form H_2O , HCl and SbCl_3 ; the chloride and the antimonate of the metal remain. The potassium salt has the formula $\text{SbCl}_5 \cdot \text{SbCl}_4(\text{OH}) \cdot 2\text{KCl}$, and forms rhombic plates; the ammonium salt has a similar formula and appearance. The calcium salt, $\text{SbCl}_5 \cdot \text{SbCl}_4 \cdot \text{OH} \cdot \text{CaCl}_2 + 9\text{H}_2\text{O}$, occurs in long prisms, and that of magnesium,—

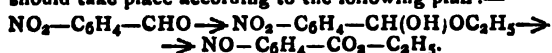


in long prisms or plates.—*Berichte*, vol. xxxiv., p. 2633.

Subchloride of Uranium.—F. Mylius and R. Dietz.—The solution of subchloride of uranium obtained by dissolving uranic acid in hydrochloric acid, after concentration on the water-bath and remaining in the desiccator, gives yellowish-green prisms which are fluorescent, very deliquescent and soluble in water, alcohol, and ether (1 portion in 0.134 part of water at 18°); the saturated aqueous solution is syrupy and denser than glass ($D=2.74$). The salt is the hydrate, $\text{UO}_2\text{Cl}_2 + 3\text{H}_2\text{O}$; its solutions have an acid reaction. If concentrated at 100° , hydrochloric acid is given off, with partial hydrolysis, and on cooling the syrupy liquid we obtain very small citron-yellow needles, non-deliquescent, very soluble in water, less soluble in alcohol than the

original salt, and having a slightly acid reaction. This new salt is $\text{UO}_2\text{HCl} + 2\text{H}_2\text{O}$ or $\text{UO}_2\text{OH} \cdot \text{Cl} + 2\text{H}_2\text{O}$; it can be compared to $\text{SO}_2\text{OH} \cdot \text{Cl}$ with regard to SO_2Cl_2 , and may be called chlorouranic acid. However, pure water does not hydrolyse it, but this result is attained by NO_3Ag , which precipitates the whole of the chlorine from the solution; the filtered liquid is yellow and astringent, it precipitates albumin, and its reaction is very slightly acid. An excess of the silver salt must not be used. In this manner we obtain a soluble, colloidal uranic acid, fairly stable at 0° , but at the ordinary temperature depositing, in a few days, a yellow body which is $\text{UO}_2\text{H}_2 + \text{H}_2\text{O}$ or UO_2H_4 , or again $\text{UO}_2(\text{OH})_4$, ortho-uranic acid; we may remark that by the addition to this formula of 1 or 2 molecules of hydrochloric acid, we obtain the two salts already described.—*Berichte*, vol. xxxiv., p. 2774.

Chemical Action of Light.—G. Ciamician and P. Silber.—In the absence of any solvent, or in the presence of a neutral solvent, such as benzene, ether, or acetone, nitrobenzaldehyde is transformed into G. Fischer's nitrosobenzoic acid in the presence of light; if heated to 180° this substance blackens, and at $205-210^\circ$ it is decomposed. In alcoholic solution *o*-nitrobenzaldehyde gives ether and the above mentioned acid; as this acid does not combine directly with alcohol it may be supposed that this addition takes place between the aldehyde and the alcohol in such a manner that the complete reaction should take place according to the following plan:—



Thus we obtain in alcoholic solution the ether of *d*-nitrosobenzoic acid in colourless crystals, fusible at $120-121^\circ$. With methylic alcohol we get the corresponding methylic ether fusing at $152-153^\circ$. With isopropylic alcohol only the free acid is produced. With the meta- and para-nitrobenzaldehydes we obtain results different to those given by the ortho-derivative; the corresponding nitroso acids are not formed, and the aldehyde remains for the greater part untransformed.—*Berichte*, vol. xxxiv., p. 2040.

Separation and Estimation of small Quantities of Potassium in Saline Mixtures.—Van Leent.—When salts of potassium are mixed with a large excess of other salts, particularly those of sodium, which is the case with sea-water, their separation by means of chloride of platinum becomes expensive and uncertain, as is also their separation in the form of perchlorate. In such a case it is advisable to make use of cobaltinitrite of sodium, as recommended by M. de Koninck. The sample being first concentrated and freed from the greater part of the lime and magnesia present, by means of carbonate of soda, the reagent is added, and after standing for six or seven hours at about 50° , the solution is filtered. The potassium in the cobaltinitrite is then estimated in the form of perchlorate or chloroplatinate. In the former case, which is the most to be recommended, the precipitate is dissolved with the ash of the filter, in hydrochloric acid, and treated with perchloric acid, in the usual manner. In the latter case, the cobalt salt is calcined gently, and the oxide washed to extract the nitrite of potassium, which is transformed into chloride. This method, applied to a sample of water from the coast of Holland, gave as result:—0.6245 grm. of chloride of potassium per litre, with 15 grms. of chloride of sodium and 5 grms. of sulphate of magnesia.—*Zeit. Anal. Ch.*, vol. xl., p. 569.

MEETINGS FOR THE WEEK.

- MONDAY, 24th.—Society of Arts, 8. (Cantor Lectures). "The Future of Coal-gas and Allied Illuminants," by Prof. Vivian B. Lewis.
- WEDNESDAY, 26th.—Society of Arts, 8. "Le Tunnel du Simplon, et la Nouvelle Ligne de Chemin de fer Directe Anglo-Italienne pour l'Orient," by Dr. Gustave Goegg, Professor of Technology at the High School of Commerce, Geneva.

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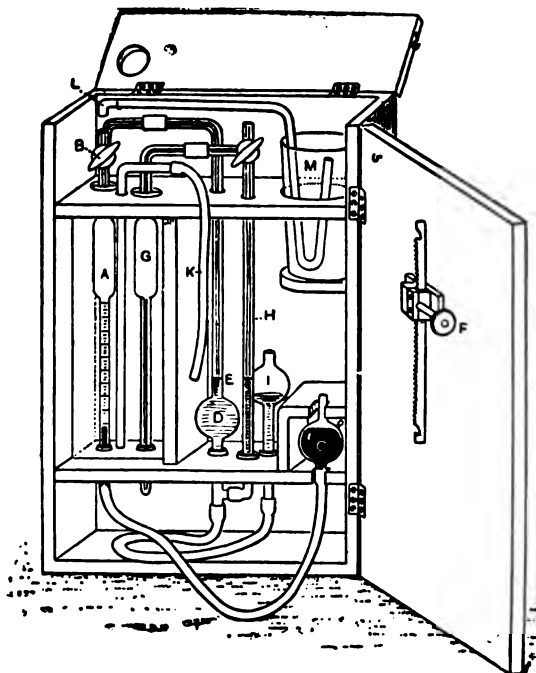
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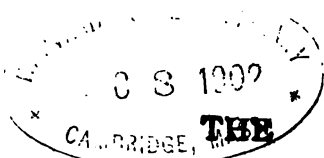
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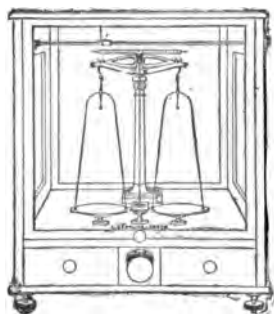
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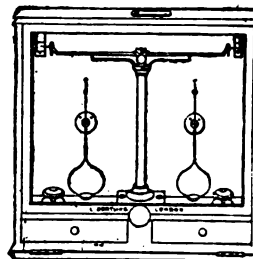


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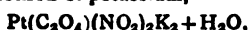
# THE CHEMICAL NEWS.

VOL. LXXXVI., No. 2244.

## THE COMPLEX SALTS OF PLATINUM. REACTIONS OF THE PLATO-OXALONITRITES.

By M. VĚZES.

### PLATO-OXALONITRITE of potassium,—

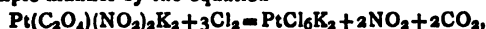


described in a previous paper (*Bull. Soc. Chim.*, Series 3, vol. xxi., p. 481), resembles various other complex salts of platinum in its reactions that I am about to describe now.

#### I. Action of Chlorine, Bromine, and Iodine.

Chlorine does not appear to form any combination with this salt analogous to that which it gives with the platonitrite of potassium, at least not when working in the wet way. In fact, when we pass a current of chlorine through a solution of the oxalonitrite, kept warm so as to prevent the deposition of the salt, which is very slightly soluble in the cold, no crystalline deposit is observed, while under the same conditions, the platonitrite gives, first of all, an abundant deposit of the plati-dichloronitrite,  $\text{Pt}(\text{NO}_2)_4\text{Cl}_2\text{K}_2$ .

Quite on the contrary, however, the solution undergoes a reaction during the passage of the chlorine, which has the effect of transforming, at least partially, the oxalonitrite into more soluble products, for, when left to stand in the cold it does not give any deposit, in spite of the concentration it has undergone during the operation. It is necessary to concentrate it further to obtain a final deposit; this consists of chloroplatinate mixed with a little unattacked oxalonitrite. The reaction of the chlorine on the oxalonitrite thus appears to be expressed in a simple manner by the equation—

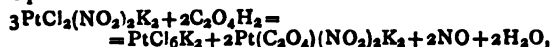


unless it forms intermediate products, such as the plati-dichloronitrite, which, it would appear, might be formed according to the equation—



Thus chlorine does not appear to be able to displace the carbon groups of the oxalonitrite without at the same time displacing the nitrogen groups. On the other hand, oxalic acid is able to displace the chlorine from a chloronitrated salt without, at the same time, displacing the whole of the nitrogen; in fact, if we act with an excess of oxalic acid on a solution of plati-dichloronitrite of potassium, a part of this salt is transformed into chloroplatinate with the evolution of nitrous fumes, but the greater portion passes into the state of oxalonitrite, thus keeping the whole of the nitrogen it originally contained.

This transformation can be represented by the equation—

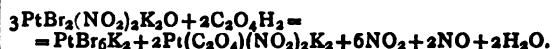


and thus would constitute a means for the preparation of the oxalonitrite from the plati-dichloronitrite of potassium, if it was not so difficult to separate the product obtained from the chloroplatinate with which it crystallises.

\* We may remark here that this fact makes untenable the hypothesis encouraged by certain other reactions, according to which plati-oxalonitrite of potassium would be a product of addition, dissociable by water, of the platonitrite and plati-oxalate of potassium; if this were the case, the solution of this salt would contain some platonitrite, and would give this characteristic reaction with chlorine, which, however, does not take place. The hypothesis in question, therefore, should be rejected; further, it is rendered very improbable by the ease with which the plati-oxalonitrite is formed in a solution containing a notable excess of one or the other of these two components.

Bromine gives with plati-oxalonitrite of potassium a reaction analogous to that of chlorine; it transforms the plati-oxalonitrite into bromoplatinate without the formation of either products of addition or intermediate products, such as the plati-dibromonitrite,  $\text{PtBr}_2(\text{NO}_2)_2\text{K}_2$ . We have simply:— $\text{Pt}(\text{C}_2\text{O}_4)_2\text{K}_2 + 3\text{Br}_2 = \text{PtBr}_6\text{K}_2 + 2\text{NO}_2 + 2\text{CO}_2$ .

Inversely, the action of oxalic acid on the bromonitrated salts of platinum gives a mixture of bromoplatinate and oxalonitrite. For example, with the plati-dibromonitrite we have:—



Iodine gives analogous results, but less distinctly on account of a possible deposit of platinic iodide which here replaces the bromoplatinate or the chloroplatinate, as well as the reduction of the salt to the state of metallic platinum by the alcohol used as a solvent. Here, again, it is not possible to obtain the plati-diiodonitrite,  $\text{PtI}_2(\text{NO}_2)_2\text{K}_2\text{O}$ , from the oxalonitrite; the inverse reaction, on the other hand, is easy.

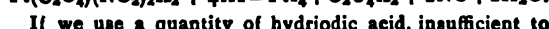
#### II. Action of Hydrochloric, Hydrobromic, and Hydriodic Acids.

Hydrochloric acid transforms plati-oxalonitrite of potassium into chloroplatinate without the formation of intermediate products, such as the plati-dichloronitrite. If we use less than six molecules of acid for each molecule of the salt, we obtain by the evaporation of the greenish-yellow liquid formed, a mixture of chloroplatinate and unattacked oxalonitrite, and, at the same time, crystals of oxalic acid, which, with the nitrous fumes given off during the operation, form the secondary products of the reaction. This reaction can be represented by the equation—



Hydrobromic acid causes an analogous transformation, and gives the bromoplatinate,  $\text{PtBr}_6\text{K}_2$ .

Hydriodic acid gives a reaction differing slightly from the preceding ones, on account of the great stability and insolubility of platinic iodide,  $\text{PtI}_4$ . It is, in fact, this body which is precipitated as an abundant black deposit when we heat a solution of plati-oxalonitrite with hydriodic acid. At the same time, nitrous fumes are given off, and the solution contains oxalate of potassium:—



If we use a quantity of hydriodic acid, insufficient to precipitate all the platinum in the form of iodide, the filtered solution, after evaporation and cooling, leaves unattacked oxalonitrite. Thus, here again, it is not possible to detect the formation of intermediate products, such as the plati-diiodonitrite.

#### III. Action of Ammonia.

Ammonia gives immediately a very abundant white precipitate with solutions of the plati-oxalonitrates, as with all the other mixed nitrated salts containing at least two atoms of nitrogen for each atom of platinum; this precipitate has, under the microscope, the appearance of a mass of very fine colourless needles; it is slightly soluble in boiling water, and less so in cold water. When washed, dried, and heated to below a red heat, this body detonates with violence without forming any carbonic gas, or leaving any other residue than platinum free from soluble matters.

These characteristics, as also the estimation of the platinum by calcination with sulphuric acid (calculated, 60.67; found, 60.76), enables us to recognise in this body the nitrite of platamine,  $\text{Pt}(\text{NO}_2)_2(\text{NH}_3)_2$ , which was described by Lang (*Journ. f. Prakt. Ch.*, vol. lxxxiii., p. 418), and which is formed in this case according to the equation—



## IV. Action of the Metallic Salts.

Plato-oxalonitrite of potassium gives particularly interesting reactions with most of the metallic salts. We have already (*Bull. Soc. Chim.*, Series 3, vol. xxv., p. 157) examined the case in which we react on this body with chloride of barium, strontium, or calcium; we propose now to examine the case of the salts of silver, copper, nickel, lead, and magnesium.

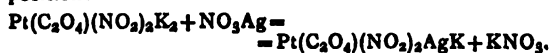
**Salts of Silver.**—Nitrate of silver, with solutions of plato-oxalonitrite of potassium, gives an immediate deposit of very fine colourless needles, very slightly soluble, especially in the cold; in fact, they are only soluble in about 1000 times their weight of cold water and in 50 times their weight of boiling water. Heated to about 110°, they lose their water of crystallisation; towards 250° they decompose, forming bubbles, and giving off binoxide of nitrogen and carbonic acid gas, at the same time leaving a black residue formed of platinum, silver, and nitrate of potassium. The crystallographical examination, made by M. Dufet, gave the following results:—

Long prisms, not more than 2 m.m. in thickness, with the facets  $A_2$  (210) and  $g_1$  (010), terminated by a perfect cleavage following the base. Clinorhombic system—

$$a:b:c = 0.8820:1:x? \\ \beta = 87^\circ 52'.$$

The extinction in  $g_1$  occurs at 6° from the vertical axis in the acute angle of the axes. The plane of the optical axes is perpendicular to  $g_1$ ; the axes seen in  $g_1$  do not extend into the air. In bromised naphthaline of  $n_D^{20} 1.6578$  index, they form an angle of  $2H = 96^\circ 22'$  round the normal to  $g_1$  in the plane 6° from the vertical axis.

Analysis gives these crystals the following formula:— $Pt(C_2O_4)(NO_2)_2AgK + H_2O$ ; thus it is an argento-potassic plato-oxalonitrite resulting from the partial double decomposition:—



Below are the analytical results found for the salt thus obtained:—

I. 1.0745 grms. of material, heated to 110°, lost 0.0350 gm. of water. The dry product, heated to about 300°, lost 0.2413 gm. of carbonic acid and nitric oxide, leaving a residue of 0.7982 gm. This residue, treated with warm water, gave up 0.1897 gm. of nitrate of potassium, containing 0.0734 gm. of potassium; washings with dilute warm nitric acid removed 0.2073 gm. of metallic silver, leaving a residue of 0.4012 gm. of platinum.

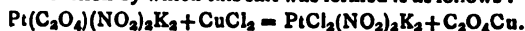
II. 0.5922 gm. of material, heated to about 300°, left a residue of 0.4403 gm., from which were separated 0.1055 gm. of nitrate of potassium containing 0.0409 gm. of potassium, 0.1134 gm. of silver, and 0.2214 gm. of platinum.

III. 0.5868 gm. of material, heated to redness with tungstic acid and copper, gave 23.6 c.c. of nitrogen at 0° and 760 m.m., and 52 c.c. of carbonic gas at 0° and 760 m.m., containing 0.0279 gm. of carbon.

|                              | Calculated. |        | Found. |       |      |
|------------------------------|-------------|--------|--------|-------|------|
|                              | I.          | II.    | I.     | II.   | III. |
| Pt .. .. .                   | 194.8       | 36.08  | 37.34  | 37.39 | —    |
| Ag .. .. .                   | 107.9       | 19.99  | 19.29  | 19.15 | —    |
| K .. .. .                    | 39.1        | 7.25   | 6.83   | 6.50  | —    |
| 2C .. .. .                   | 24.0        | 4.45   | —      | —     | 4.75 |
| 2N .. .. .                   | 28.1        | 5.20   | —      | —     | 5.05 |
| 8O .. .. .                   | 128.0       | 23.70  | —      | —     | —    |
| H <sub>2</sub> O .. .. .     | 18.0        | 3.33   | 3.26   | —     | —    |
| $Pt(C_2O_4)(NO_2)_2AgK.H_2O$ | 539.9       | 100.00 | —      | —     | —    |

**Salts of Copper.**—If we mix two solutions of plato-oxalonitrite of potassium and cupric chloride in equimolecular proportions, the blue liquid obtained, which is first of all limpid, becomes cloudy and soon deposits an abundant bright blue precipitate having a crystalline appearance under the microscope. After boiling for some moments to complete the precipitation, if we separate the

precipitate by filtration and wash it carefully, we find by qualitative analysis that it consists of pure oxalate of copper, and by electrolytic estimation that it contains practically all the cupric chloride used; in an experiment made with 20 grms. of oxalonitrite and 7.2 grms. of hydrated cupric chloride,  $CuCl_2 \cdot 2H_2O$ , containing 2.7 grms. of copper, the electrolysis of the precipitated oxalate re-dissolved in an excess of oxalate of ammonium, gave 2.6 grms. of copper. The liquid, filtered after the precipitation of the copper as oxalate, is of a golden-yellow colour. When strongly concentrated by slow evaporation it gives yellow crystals of plato-dichloronitrite of potassium,  $PtCl_2(NO_2)_2K_2$  (platinum calculated, 44.67; found, 44.79—chloride of potassium calculated, 34.21; found, 34.05—nitrogen calculated, 6.44; found, 6.29). The reaction by which this salt was formed is as follows:—



Thus, we see that the action of chloride of copper on plato-oxalonitrite of potassium gives the same product as the carefully managed action of hydrochloric acid on the platonitrite. This conclusion can be generalised, as it is easy to make certain by reading on the plato-oxalonitrite with another salt of copper such as the sulphate or nitrate.

Certain strong acids, particularly sulphuric and nitric acids, react on the platonitrite under the influence of heat, giving an acid product crystallised in the form of red needles; this is the acid triplatohexanitrite of potassium,  $Pt_3O(NO_2)_6K_2H_4 + 3H_2O$  (*Comptes Rendus*, vol. cxvi., pp. 99, 160, and 185).

Now, if we mix equivalent solutions of plato-oxalonitrite and sulphate or nitrate of copper, we obtain first of all a bright blue, crystalline precipitate, containing all the copper in the form of oxalate, and the filtrate after concentration deposits an abundance of red needles of this acid triplatohexanitrite. Thus we can say in a general way that the action of a copper salt on plato-oxalonitrite of potassium gives the same product as the carefully managed action of the acid of this salt on the platonitrite.

**Salts of Nickel, Lead, and Magnesium.**—The same rule appears to apply to the salts of most of the heavy metals. It is in this manner that chloride of nickel gives a solution of plato-dichloronitrite, with a precipitate of oxalate of nickel containing all the metal from the chloride used. Chloride of lead in the same way gives a deposit of oxalate of lead and a solution of plato-dichloronitrite of potassium. Iodide of lead, in spite of the special difficulties resulting from its slight solubility, gave with a precipitate of oxalate of lead a solution of plato-diiodonitrite of potassium,  $PtI_2(NO_2)_2K_2 + 2H_2O$ . Finally, bromide of magnesium in the same way gave with a deposit of oxalate of magnesium a solution which after concentration gave plato-dibromonitrite of potassium,  $PtBr_2(NO_2)_2K_2 + H_2O$  (platinum calculated, 35.84; found, 35.73—bromide of potassium calculated, 43.89; found 43.16—water calculated, 3.32; found, 3.70).

These reactions, which are effected in neutral solution, thus constitute a convenient means for the preparation of the mixed plato-salts of  $PtX_2(NO_2)_2K_2$ ; they furnish products purer than those obtained by the action of the hydrides on the platonitrite, admitting the tendency which all the chloro- or bromo-nitrated compounds of platinum have to form a chloroplatinate or a bromoplatinate when they are heated in an acid solution.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 18.

**Analysis of Nine Specimens of Air taken from the Galleries of Mines.**—Nestor Gréhan. —The author's experiments were conducted in the galleries of a coal mine in process of working. The most noticeable feature of the analysis is the large proportion of formene in such air. The carbonic acid varies between 1 and 1.8 per cent. The proportion of oxygen is perceptibly less than that of normal air.—*Comptes Rendus*, cxxxv., No. 18.

# THE ANALYSIS OF LITHOPONE.

By CH. COFFIGNIER.

In a previous paper (*Bull. Soc. Chim.*, vol. xxvii., p. 829) I gave the compositions of a series of lithopones of different manufactures, and the method of analysis used. At the end of this paper I drew attention to a certain number of abnormal results obtained. Among these, *one* only was due to an analytical error in the estimation of the sulphate of barium; all the others require explanation.

Since the publication of my first paper, I have succeeded in obtaining further samples, and thus confirming the first results in such a manner as to show that the results obtained are all to be depended upon. I have also analysed richer samples, containing from 40 to 50 per cent of sulphide of zinc.

I will give now the whole series of analyses which have given me results analogous to those I obtained previously.

## Specimens containing 20 to 22 per cent of Sulphide of Zinc.

|                       | E.     |       | A.    |       |
|-----------------------|--------|-------|-------|-------|
|                       | 1.     | 2.    | 1.    | 2.    |
| Moisture .. ..        | —      | 0.18  | —     | 0.12  |
| Aqueous extract ..    | —      | 0.32  | —     | 0.24  |
| Oxide of zinc .. ..   | —      | 1.50  | —     | 1.53  |
| Sulphide of zinc ..   | 21.40  | 19.34 | 25.40 | 23.47 |
| Sulphate of barium .. | 78.63  | 78.63 | 74.40 | 74.40 |
|                       | 100.03 | 99.97 | 99.80 | 99.76 |

## Specimens containing 26 per cent Sulphide of Zinc.

|                       | E.    |        |
|-----------------------|-------|--------|
|                       | 1.    | 2.     |
| Moisture .. ..        | —     | 0.30   |
| Aqueous extract ..    | —     | 0.34   |
| Oxide of zinc .. ..   | —     | 1.34   |
| Sulphide of zinc ..   | 26.75 | 24.90  |
| Sulphate of barium .. | 73.22 | 73.22  |
|                       | 99.97 | 100.10 |

## Specimens containing 29 to 30 per cent Sulphide of Zinc.

|                       | C.    |       | G.     |       |
|-----------------------|-------|-------|--------|-------|
|                       | 1.    | 2.    | 1.     | 2.    |
| Moisture .. ..        | —     | 0.14  | —      | 0.18  |
| Aqueous extract ..    | —     | 0.28  | —      | 0.14  |
| Oxide of zinc .. ..   | —     | 1.46  | —      | 2.95  |
| Sulphide of zinc ..   | 30.50 | 28.68 | 31.05  | 27.05 |
| Sulphate of barium .. | 61.10 | 69.10 | 69.54  | 69.54 |
|                       | 99.60 | 99.66 | 100.59 | 99.86 |

## Specimens containing 33 to 34 per cent Sulphide of Zinc.

|                       | A.     |        | C.    |        |
|-----------------------|--------|--------|-------|--------|
|                       | 1.     | 2.     | 1.    | 2.     |
| Moisture .. ..        | —      | 0.04   | —     | 0.20   |
| Aqueous extract ..    | —      | 0.08   | —     | 0.20   |
| Oxide of zinc .. ..   | —      | 1.98   | —     | 0.59   |
| Sulphide of zinc ..   | 34.60  | 32.01  | 37.12 | 36.40  |
| Sulphate of baryta .. | 66.03  | 66.00  | 62.73 | 62.73  |
|                       | 100.60 | 100.11 | 99.85 | 100.12 |

|                       | D.*   |       |
|-----------------------|-------|-------|
|                       | 1.    | 2.    |
| Moisture .. ..        | —     | 0.08  |
| Aqueous extract ..    | —     | 0.54  |
| Oxide of zinc .. ..   | —     | 0.65  |
| Sulphide of zinc ..   | 20.47 | 19.40 |
| Sulphate of barium .. | 79.20 | 79.20 |
|                       | 99.67 | 99.87 |

\* This sample, marked 33 to 34 per cent, was certainly badly taken. It comes in the class 20 to 22 per cent.

## Very Rich Specimens.

|                     | A.           |       | A.           |       | A.           |       |
|---------------------|--------------|-------|--------------|-------|--------------|-------|
|                     | 40 per cent. |       | 45 per cent. |       | 50 per cent. |       |
|                     | 1.           | 2.    | 1.           | 2.    | 1.           | 2.    |
| Moisture .. ..      | —            | 0.20  | —            | 0.18  | —            | 0.10  |
| Aqueous extract ..  | —            | 0.24  | —            | 0.34  | —            | 0.56  |
| Oxide of zinc .. .. | —            | 2.00  | —            | 2.00  | —            | 2.40  |
| Sulphide of zinc .. | 41.27        | 38.80 | 45.11        | 42.37 | 50.36        | 47.52 |
| Sulphate of barium  | 58.35        | 58.35 | 54.83        | 54.83 | 49.14        | 49.14 |
|                     | 99.62        | 99.59 | 99.94        | 99.72 | 99.50        | 99.72 |

All these results, obtained with samples from six different makers, show conclusively that the method of analysis is to be depended upon.

The analyses of which I am now about to speak, total up in Column 1 to the figures 96.97 to 98.83; in Column 2, from 97.99 to 98.83. In both these cases the figures obtained are inadmissible.

Now, these results were always obtained with samples from the same maker. This is the first point which attracted my attention.

Lithopone is obtained by precipitating a solution of sulphate of zinc with a solution of sulphide of barium; the precipitate obtained contains oxysulphhydrate of zinc, hydrated oxide of zinc, and sulphate of barium. The action of water on the sulphide of barium obtained by the reduction of the sulphate, gives an oxysulphhydrate of barium and hydrate of baryta. Calcination of the precipitate transforms the oxysulphhydrate into sulphide and the hydrated oxide into oxide; from which it follows that commercial lithopone is a mixture of sulphide of zinc, oxide of zinc, and sulphate of baryta. Thus it is very possible that these types giving abnormal results have not been sufficiently calcined. By taking the zinc soluble in dilute acetic acid to be  $Zn\begin{smallmatrix} SH \\ OH \end{smallmatrix}$ , instead of  $ZnO$ , we get a more likely result, as will be seen from an examination of the following figures:—

|                                                          | F (22 to 23 per cent). |       |        | F (29 to 30 per cent). |       |        |
|----------------------------------------------------------|------------------------|-------|--------|------------------------|-------|--------|
|                                                          | 1.                     | 2.    | 3.     | 1.                     | 2.    | 3.     |
| H <sub>2</sub> O .. ..                                   | —                      | 1.46  | 1.46   | —                      | 1.50  | 1.50   |
| Aqueous extract ..                                       | —                      | 0.80  | 0.80   | —                      | 0.72  | 0.72   |
| ZnO .. ..                                                | —                      | 3.54  | —      | —                      | 4.34  | —      |
| $Zn\begin{smallmatrix} SH \\ OH \end{smallmatrix}$ .. .. | —                      | —     | 5.02   | —                      | —     | 6.17   |
| ZnS .. ..                                                | 26.50                  | 22.28 | 22.28  | 31.13                  | 25.87 | 25.87  |
| SO <sub>4</sub> Ba .. ..                                 | 70.75                  | 70.75 | 70.75  | 65.95                  | 65.95 | 65.95  |
|                                                          | 97.25                  | 98.83 | 100.31 | 97.08                  | 98.38 | 100.21 |

|                                                          | F (32 to 34 per cent). |       |        | F (40 to 42 per cent). |       |        |
|----------------------------------------------------------|------------------------|-------|--------|------------------------|-------|--------|
|                                                          | 1.                     | 2.    | 3.     | 1.                     | 2.    | 3.     |
| H <sub>2</sub> O .. ..                                   | —                      | 0.86  | 0.86   | —                      | 1.00  | 1.00   |
| Aqueous extract ..                                       | —                      | 1.16  | 1.16   | —                      | 1.34  | 1.34   |
| ZnO .. ..                                                | —                      | 4.27  | —      | —                      | 5.92  | —      |
| $Zn\begin{smallmatrix} SH \\ OH \end{smallmatrix}$ .. .. | —                      | —     | 6.07   | —                      | —     | 8.28   |
| ZnS .. ..                                                | 34.81                  | 29.54 | 29.54  | 41.92                  | 34.68 | 34.68  |
| SO <sub>4</sub> Ba .. ..                                 | 62.60                  | 62.60 | 62.60  | 55.05                  | 55.05 | 55.05  |
|                                                          | 97.41                  | 98.43 | 100.24 | 96.97                  | 97.99 | 100.35 |

If the analyses in Columns 3 are a little too high, it is really because the whole of the soluble zinc is not in the form of  $Zn\begin{smallmatrix} SH \\ OH \end{smallmatrix}$ , but partly in this state and partly as ZnO.

It may be remarked that all these types, compared with the preceding ones, give a much higher proportion of moisture and ZnO.

From the point of view of the explanation I have just given, I will give the results of the analyses obtained from two samples sent to me by French makers who were endeavouring to establish the manufacture of lithopone

without calcination. The products they sent me were obtained by means of double decomposition, passing through the filter-press, and drying at about  $110^{\circ}$ .

|                                                                           | H.    |       |       | I.    |       |       |
|---------------------------------------------------------------------------|-------|-------|-------|-------|-------|-------|
|                                                                           | 1.    | 2.    | 3.    | 1.    | 2.    | 3.    |
| H <sub>2</sub> O .. .. .                                                  | —     | 2.88  | 2.88  | —     | 2.66  | 2.66  |
| Aqueous extract ..                                                        | —     | 0.48  | 0.48  | —     | 2.02  | 2.02  |
| ZnO .. .. .                                                               | —     | —     | —     | —     | 1.40  | —     |
| Zn $\begin{smallmatrix} \text{SH} \\ \text{OH} \end{smallmatrix}$ .. .. . | —     | —     | 2.36  | —     | —     | 1.95  |
| ZnS .. .. .                                                               | 28.43 | 26.60 | 26.60 | 27.95 | 26.36 | 26.36 |
| SO <sub>4</sub> Ba .. .. .                                                | 67.40 | 67.40 | 67.40 | 66.70 | 66.70 | 66.70 |
|                                                                           | 95.83 | 99.02 | 99.72 | 94.65 | 99.14 | 99.69 |

We see that here, as in the preceding cases, the analysis is only satisfactory when we admit that the zinc soluble in dilute acetic acid is in the state of  $\text{Zn}\begin{smallmatrix} \text{SH} \\ \text{OH} \end{smallmatrix}$ . To obtain

an experimental proof of this theory I precipitated a solution of sulphide of barium by chloride of zinc. The washed precipitate, treated with acetic acid at 1 per cent, gave a solution precipitable with sulphuretted hydrogen. During the action of the dilute acetic acid the odour of sulphuretted hydrogen was distinctly observable. All these characteristics were observed when attacking lithopones which had given abnormal analytical results with dilute acetic acid. Taking these observations altogether, it appears to me to be quite evident that the presence of the oxysulphhydrate of zinc will account for the anomalies observed in the analysis of some of the samples.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 18.

## CONTRIBUTIONS TO OUR KNOWLEDGE OF YTTERBIUM.\*

By ASTRID CLEVE.

(Continued from p. 249).

### III. COMPOUNDS OF YTTERBIUM.

#### Ytterbium Oxide, Yb<sub>2</sub>O<sub>3</sub>.

FIRST examined and described by Nilson, according to whom it has the high specific gravity 9.175. When perfectly pure the substance is colourless. But probably the least traces of thulium are sufficient to colour the oxide, as the preparations after very strongly heating mostly have a yellow or brownish tinge. The oxide is slowly attacked by acids in solutions, as well as at a red heat. It is quite insoluble in weak acids such as acetic acid.

According to a communication from Dr. Euler, the earth is not radio-active.

#### Ytterbium Chloride, YbCl<sub>3</sub> + 6H<sub>2</sub>O.

The salt is very soluble and deliquescent. It may be precipitated in crystals by dissolving the oxide in concentrated hydrochloric acid, and saturating with hydrochloric acid gas; it then forms beautiful, clear, tapering prisms. It does not part with any of its water to sulphuric acid or lime, or when heated to  $70^{\circ}$ . At  $100^{\circ}$  slow decomposition takes place, and on strongly heating the chlorine is given up altogether.

#### Analyses.

1. 0.9082 grm. of the substance dried at  $70^{\circ}$  gave on ignition 0.4631 grm. Yb<sub>2</sub>O<sub>3</sub>.
2. 0.7313 grm. of the substance dried over CaO gave 0.8141 grm. AgCl.

|                           | Calculated for<br>YbCl <sub>3</sub> + 6H <sub>2</sub> O, in per cent. |        | Found. |       |
|---------------------------|-----------------------------------------------------------------------|--------|--------|-------|
|                           | I.                                                                    | II.    | I.     | II.   |
| Yb .. .. .                | 173.1                                                                 | 44.66  | 44.78  | —     |
| Cl <sub>3</sub> .. .. .   | 106.35                                                                | 27.44  | —      | 27.52 |
| 6H <sub>2</sub> O .. .. . | 108.12                                                                | 27.90  | —      | —     |
|                           | 387.57                                                                | 100.00 |        |       |

\* From the *Zeitschrift für Anorganische Chemie*, xxxii.

#### Specific Gravity and Molecular Volume.

|                                         |       |
|-----------------------------------------|-------|
| 1. 1.2550 grms., specific gravity .. .. | 2.570 |
| 2. 1.1056 " " " " .. ..                 | 2.580 |
| Mean .. .. .                            | 2.575 |
| Molecular volume .. ..                  | 150.5 |

#### Ytterbium Oxychloride, YbOCl.

A substance approximately of this composition was obtained by dissolving the chloride in water and heating in a current of hydrochloric acid gas, first to dryness, and then to a red heat. At some time a white hygroscopic residue was obtained, and was analysed.

1. 0.6237 grm. of the substance, after heating before the blowpipe, gave 0.5338 grm. Yb<sub>2</sub>O<sub>3</sub>.
2. 0.8894 grm. of the substance gave 0.6819 grm. AgCl.

|            | Calculated for<br>YbOCl, in per cent. | Found. |       |
|------------|---------------------------------------|--------|-------|
|            |                                       | I.     | II.   |
| Yb .. .. . | 77.08                                 | 75.17  | —     |
| Cl .. .. . | 15.79                                 | —      | 18.95 |

The analysis shows that the formation of the oxychloride was not quite completed, probably because it had not been heated sufficiently.

#### Ytterbium Bromide, YbBr<sub>3</sub> + 8H<sub>2</sub>O.

This was prepared in the same way as the preceding salt, which it resembles, though it is still more deliquescent. The solution dries very slowly over lime, giving crystals containing 8 molecules of water.

#### Analysis of the Pressed Salt.

1. 1.0463 grms. of the substance gave 1.0518 grms. AgBr.
2. 1.4707 grms. of the substance gave 1.4797 grms. AgBr.
3. 0.7621 grm. of the substance gave 0.2686 grm. Yb<sub>2</sub>O<sub>3</sub>.

|                           | Calculated for<br>YbBr <sub>3</sub> + 8H <sub>2</sub> O, in per cent. |        | Found. |       |       |
|---------------------------|-----------------------------------------------------------------------|--------|--------|-------|-------|
|                           |                                                                       |        | I.     | II.   | III.  |
| Yb .. .. .                | 173.1                                                                 | 31.07  | —      | —     | 30.95 |
| Br <sub>3</sub> .. .. .   | 239.88                                                                | 43.06  | 42.78  | 42.81 | —     |
| 8H <sub>2</sub> O .. .. . | 144.16                                                                | 25.87  | —      | —     | —     |
|                           | 557.14                                                                | 100.00 |        |       |       |

The bromides of yttrium and erbium crystallise possibly with the same amount of water (cf. Cleve and Höglund, *Bull. Soc. Chim.*, 1872, xviii., 196).

#### Ytterbium Iodate, Yb<sub>3</sub>IO<sub>6</sub> + 6H<sub>2</sub>O (Dried over Sulphuric Acid).

It is produced in the form of a white amorphous precipitate when solutions of iodic acid and ytterbium acetate are mixed. When dried, it gives a snow-white powder.

#### Analysis.

- 0.9863 grm. of the substance dried in the desiccator gave up 0.0365 grm. of water at  $100^{\circ}$ .
- 0.3268 grm. of the substance gave, after reduction with SO<sub>2</sub>, 0.2852 grm. AgI.

|                           | Calculated for<br>Yb <sub>3</sub> IO <sub>6</sub> + 6H <sub>2</sub> O, in per cent. | Found.                                   |     |
|---------------------------|-------------------------------------------------------------------------------------|------------------------------------------|-----|
|                           |                                                                                     | I.                                       | II. |
| I .. .. .                 | 47.23                                                                               | 47.15                                    | —   |
| 2H <sub>2</sub> O .. .. . | 4.27                                                                                | 3.70 (loss of weight at $100^{\circ}$ ). | —   |

Thus 4 molecules of water remained in combination above  $100^{\circ}$ .

#### Ytterbium Periodate, YbIO<sub>5</sub> + 2H<sub>2</sub>O (Dried over Sulphuric Acid).

A white hygroscopic powder, obtained from ytterbium acetate and periodic acid. After having been dried in the sulphuric acid desiccator, it suffers no loss of weight at  $100-115^{\circ}$ .

Analysis.

0.4521 grm. of the substance dried over sulphuric acid gave, after reduction with  $\text{SO}_2$ , 0.2556 grm. AgI.  
0.2134 grm. of  $\text{Yb}_2\text{O}_3$  were precipitated from the filtrate by means of ammonia.

| Calculated for<br>$\text{YbIO}_4 + 2\text{H}_2\text{O}$ , in per cent. |        |       | Found. |
|------------------------------------------------------------------------|--------|-------|--------|
| Yb ..                                                                  | 173.1  | 41.61 | 41.46  |
| I ..                                                                   | 126.85 | 30.50 | 30.55  |
| $\text{O}_5$ ..                                                        | 80     | 19.23 | —      |
| $2\text{H}_2\text{O}$ ..                                               | 36.04  | 8.66  | —      |
| 415.99                                                                 |        |       | 100.00 |

Corresponding periodates of yttrium (Cleve, *Bull. Soc. Chim.*, 1874, xxi., 345) and samarium (Cleve, "Contributions to the Knowledge of Samarium," *Nova Acta Reg. Soc. Sc. Ups.*, Ser. III., p. 16)—the latter, however, with  $4\text{H}_2\text{O}$  at  $100^\circ$ —are known.

Ytterbium-platinum Chlorides.

1.  $2\text{YbCl}_3 \cdot \text{PtCl}_4 + 22\text{H}_2\text{O}$ .

Crystallises from the mixture of the solutions of ytterbium chloride and platinum chloride (the latter in excess) in large transparent rhombic tables of reddish brown colour. The crystals deliquesce in damp air, but crumble in the desiccator, the colour changing to light yellow. At  $100^\circ$ , half the water of crystallisation is given up; below this temperature, the salt dissolves in its water of crystallisation. Its reaction is neutral.

Analysis of the Pressed Substance.

- 0.5424 grm. of the substance gave up 0.0839 grm. of water at  $100^\circ$ , and, after ignition, gave a residus of 0.3524 grm.  $\text{Yb}_2\text{O}_3 + \text{Pt}$ , of which 0.0820 grm. was platinum.
- 0.5552 grm. of the substance gave 0.3553 grm.  $\text{Yb}_2\text{O}_3 + \text{Pt}$ , of which 0.2740 grm. was  $\text{Yb}_2\text{O}_3$  and 0.0813 grm. platinum.
- 0.5557 grm. of the substance gave 0.6354 grm. AgCl.

| Calculated for<br>$2\text{YbCl}_3 \cdot \text{PtCl}_4 + 22\text{H}_2\text{O}$ ,<br>in per cent. |        |       | Found.                                                   |       |       |
|-------------------------------------------------------------------------------------------------|--------|-------|----------------------------------------------------------|-------|-------|
|                                                                                                 |        |       | I.                                                       | II.   | III.  |
| Yb <sub>2</sub> ..                                                                              | 346.2  | 26.80 | 27.33                                                    | 27.07 | —     |
| Pt ..                                                                                           | 194.8  | 15.08 | 15.1                                                     | 14.7  | —     |
| Cl <sub>10</sub> ..                                                                             | 354.5  | 27.44 | —                                                        | —     | 27.20 |
| $22\text{H}_2\text{O}$ ..                                                                       | 396.44 | 30.68 | 15.47 (loss of $\text{H}_2\text{O}$<br>at $100^\circ$ ). | —     | —     |
| 1291.94                                                                                         |        |       | 100.00                                                   |       |       |
| $\text{Yb}_2\text{O}_3 + \text{Pt}$ ..                                                          | 64.18  | 64.98 | 64.00                                                    |       |       |

2.  $2\text{YbCl}_3 \cdot \text{PtCl}_4 + 35\text{H}_2\text{O}$ .

Another preparation gave on analysis the above quantity of water.

- 0.6240 grm. of the pressed substance gave 0.3400 grm.  $\text{Yb}_2\text{O}_3 + \text{Pt}$ , of which 0.2602 was  $\text{Yb}_2\text{O}_3$  and 0.0798 grm. was Pt.

| Calculated for<br>$2\text{YbCl}_3 \cdot \text{PtCl}_4 + 35\text{H}_2\text{O}$ ,<br>in per cent. |       |       | Found. |
|-------------------------------------------------------------------------------------------------|-------|-------|--------|
| Yb <sub>2</sub> ..                                                                              | 346.2 | 22.69 | 22.76  |
| Pt ..                                                                                           | 194.8 | 12.76 | 12.8   |
| Cl <sub>10</sub> ..                                                                             | 354.5 | 23.23 | —      |
| $35\text{H}_2\text{O}$ ..                                                                       | 630.7 | 41.32 | —      |
| 1526.2                                                                                          |       |       | 100.00 |
| $\text{Yb}_2\text{O}_3 + \text{Pt}$ ..                                                          | 54.33 | 54.49 | —      |

Thus both the above salts do not belong to the ordinarily occurring type of platinum double chlorides of rare earths,  $\text{R}^{III}\text{Cl}_3 \cdot \text{PtCl}_4 + n\text{H}_2\text{O}$ ; where  $\text{R}^{III}$  may be praseodymium (v. Schéele, "Om Praseodym, &c.," *Inaug. Diss.*, Upsala, 1900, p. 47), samarium (Cleve, "Contributions to the Knowledge of Samarium," p. 10), gadolinium (Benedicks,

*Zeit. Anorg. Chem.*, xxii., 404), or erbium (Cleve, *Bull. Soc. Chim.*, 1874, xxi., 345), but contain  $2\text{R}^{III}\text{Cl}_3$  to each  $\text{PtCl}_4$ . The ytterbium salt has the composition  $4\text{YCl}_3 \cdot 5\text{PtCl}_4 + 51\text{H}_2\text{O}$  (Cleve and Nilson, *Bull. Soc. Chim.*, 1877, xxvii., 209).

Acid Ytterbium Platinum Bromide,  
 $\text{YbBr}_3 \cdot 3\text{H}_2\text{Br}_6\text{Pt} + 30\text{H}_2\text{O}$ .

It was prepared in the same way as the platinum double chloride, and forms dark red rhombic tables, which are exceedingly deliquescent. The salt has an acid reaction, and decomposes carbonates. A neutral double bromide could not be isolated in this way.

Analysis of the Pressed Substance.

- 0.8078 grm. of the salt gave 0.2540 grm.  $\text{Yb}_2\text{O}_3 + \text{Pt}$ , of which 0.0951 grm. was  $\text{Yb}_2\text{O}_3$  and 0.1589 grm. Pt.
- 0.3438 grm. of the substance gave 0.4494 grm. AgBr.

| Calculated for<br>$\text{YbBr}_3 \cdot 3\text{H}_2\text{Br}_6\text{Pt} + 30\text{H}_2\text{O}$ ,<br>in per cent. |         |       | Found. |       |
|------------------------------------------------------------------------------------------------------------------|---------|-------|--------|-------|
|                                                                                                                  |         |       | I.     | II.   |
| Yb ..                                                                                                            | 173.1   | 5.80  | 6.46   | —     |
| H <sub>6</sub> ..                                                                                                | 6.06    | 0.20  | —      | —     |
| Pt <sub>3</sub> ..                                                                                               | 584.4   | 19.59 | 19.66  | —     |
| $\text{Br}_{21}$ ..                                                                                              | 1679.16 | 56.29 | —      | 55.63 |
| $30\text{H}_2\text{O}$ ..                                                                                        | 540.6   | 18.12 | —      | —     |
| 2983.32                                                                                                          |         |       | 100.00 |       |

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, November 6, 1902.

Professor McLEOD, F.R.S., Vice-President, in the Chair.

THE CHAIRMAN referred to the great loss which the Society had sustained since its last meeting by the deaths of Sir Frederick Abel and Dr. J. H. Gladstone, two of its Past Presidents, who had been for more than fifty years Fellows of the Society.

Messrs. Garle, Hann, Pollitt, Gill, Moore, and Parkes were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. John Frederick Alder, 13, Priestwood Mansions, Highgate, N.; Alfred Edward Beanes, Moatlands, Pad-dock Wood; Herbert Blair, No. 3, General Hospital, Kroonstad; John A. H. Brincker, B.A., M.B., 7, St. Mary's Terrace, Paddington, W.; Vernon Seymour Bryant, B.A., Camborne, Cornwall; Thomas Burnell Carmichael, Parton, Whitehaven; Charles Cockle, Peter Symonds School, Winchester; James Justinian Drought, Johannesburg; Walter Henry Edwards, The Grammar School, Wellingborough; Wilfrid Russell Grimwade, B.Sc., Melbourne; Edward Gumersall, Atbara, Sildcup; David V. Hollingworth, Aldred House, Crescent, Salford; Alfred Holt, jun., B.A., Crofton, Aigburth, Liverpool; Clements F. V. Jackson, A.Inst.C.E., Brisbane; John Petty Leather, 17, Carlton Road, Burnley; Frank A. Lidbury, M.Sc., Owens College, Manchester; Ernest Liotard, 2, Rue de France, Nice; Thomas Stratford Logan, 52, Kirkgate, Leeds; Douglas A. MacCullum, Shawlands, Glasgow; William Mann, B.Sc., Fonnereau Villa, Dartford; John Marsh, 29, High Street, Maidstone; Joseph William Mellor, D.Sc., Owens College, Man- chester; John Price Millington, B.A., Christ's College, Cambridge; John Phelps, B.A., Trinity College, Oxford; George A. P. Ross, M.B., B.Ch., 20, Westhall Gardens, Edinburgh; John William Sampson, Helpringham, Heckington; Duncan Simpson, 10, Brynmill Crescent,

Swansea; Robert Walter Sindall, 80, Manor Road, Brockley, S.E.; John Seabury Smythe, B.Sc., Ph.D., 143, Smithdown Lane, Liverpool; Walter F. Sutherland, Ph.D., Agricultural College, Holmes Chapel; George Sutherland Thomson, Government Offices, Adelaide; Henry Letheby Tidy, B.A., New College, Oxford; William Carter White, 113, Gower Street, W.C.; Lyndon Wilson, 66, Castle Gate, Newark-on-Trent.

Of the following papers, those marked \* were read:—

\*145. "*The Specific Heats of Gases.*" By H. CROMPTON. It was shown that the molecular heat of any gas at the absolute temperature  $T$  may be calculated with the help of a formula of the type  $A + BT$ . In this formula  $A$  has the same value for all gases, and depends on changes in the movements of the molecules of the gas as a whole.  $B$  is proportional to  $V \times \log n$ , where  $V$  is the volume of the molecule, and  $n$  is the number of atoms in the molecule.  $BT$  depends on interatomic changes taking place within the molecule. If for  $V$  the molecular volume at the normal boiling point of the liquid  $M\theta_b$  is taken, the molecular heat at constant pressure  $C_p = 4.935 + 0.000245 M\theta_b T \log n$  (or if common logarithms are used the coefficient is 0.000564). The molecular heats calculated from this formula are shown to be in fair agreement with those observed. The ratio  $k$  of the two specific heats can also be calculated with the aid of this formula.

#### DISCUSSION.

Dr. TRAVERS remarked that it would be interesting to know how closely the formula would reproduce the values of the specific heats of the commoner gases, which had been determined with great accuracy and over a considerable range of temperature by Regnault, Witkowski, and others. The agreement between the observed and calculated values in the cases cited by the author was, however, remarkable.

Dr. PHILIP pointed out that the values of  $C_p$  given by the two formulæ (Le Chatelier's and that of the author) would, owing to the difference in the numerical constants chosen, be more divergent the lower the temperature. On this account, a study of the applicability of the proposed formula at low temperatures would be specially interesting.

Mr. CROMPTON, in reply, admitted that at low temperatures the results given by the formula did not agree with the observed so well as those obtained at higher temperatures. In explanation of this, he pointed out that the formula was only supposed to hold for an ideal gas, no account being taken of the effect on the specific heat of possible attractions between the molecules. If such attractions existed, they would have a greater influence on the specific heat at low temperatures, and this the nearer the gas was to its point of liquefaction, than under ordinary conditions. No doubt a term should be introduced to allow for the effect of molecular attractions, but at present he was unable to make any suggestion with reference to the character of this correction.

\*146. "*The Action of Nitric Acid on Bromophenolic Compounds.*" By W. ROBERTSON.

The action of nitric acid on bromophenols and their derivatives was studied in order to determine the conditions under which the bromine atom is replaceable by nitroxyl. It has been found that if the hydrogen of the hydroxyl group be replaced by either the methyl or acetyl radicle, no displacement of bromine by the nitro-group occurs when these substances are acted on by nitric acid under similar conditions. The bromohydroxybenzoic acids and their methoxy- and acetoxy-derivatives were also prepared, and it was found that with regard to them the same rule applied.

The following compounds were described:—2:6-dibromo-4-acetyl aminophenol, needles, m. p. 185–186° (Hözl. *Journ. Prakt. Chem.*, 1886, [ii.], xxxii., 68, gives 173–174°; Friedländer and Stange, *Ber.*, 1893, xxvi., 2262, 185°); 2-nitro 6-bromo-4-acetylaminophenol, yellow

needles melting at 230°; 2-nitro-6-bromo-4-benzoylamino-phenol, yellow needles, m. p. 247°; 2:3:6-tribromo-4-acetylaminophenol, white needles, m. p. 224° with decomposition; 2:6-dibromo-4-anisidine, white glistening leaflets, m. p. 66° (Stadel, *Annalen*, 1883, ccxvii., 70, does not give melting-point), and its acetyl and benzoyl derivatives which crystallise in needles melting at 206° and 180° respectively; acetyl-3:5-dibromosalicylic acid, m. p. 156°; acetyl-5-bromosalicylic acid, m. p. 168°; acetyl-3:5-dibromo-p-oxybenzoic acid, m. p. 207°; ethyl ester of 3:5-dibromo-p-oxybenzoic acid, needles, m. p. 99°; acetyl-3-bromo-p-oxybenzoic acid, m. p. 155°; a dibromo-m-oxybenzoic acid, m. p. 202°; 2-bromo-4:6-dinitro-3-oxybenzoic acid, a yellow substance, m. p. 217–218°.

#### DISCUSSION.

Mr. W. A. DAVIS pointed out that although Mr. Robertson had been unable to isolate intermediate products of nitration in the case of the bromo-phenols he had dealt with, such substances had been obtained by Armstrong and Rossiter in the form of nitrobromo-ketonaphthalenes (*Proc.*, 1891, vii., 89). He himself had for several years been working with this class of compounds, and had found that their stability depends largely on the proportion of bromine which they contain. Thus, whilst the nitro-keto-compound of  $\beta$ -naphthol is so unstable that it cannot be isolated, and the keto-derivatives of bromo-, dibromo-, and tribromo- $\beta$ -naphthol are stable only at low temperatures, those formed from the tetrabromo- and pentabromo-naphthols can usually be kept for several days in the dry state without undergoing change. Moreover, whilst the lower brominated keto-compounds are readily reduced to the corresponding bromonitro-naphthols by sodium sulphite, those derived from the tetrabromo- $\beta$ -naphthols are reducible only by concentrated hydriodic acid.

\*147. "*3:5-Dichloro-o-xylene and 3:5-Dichloro-o-phthalic Acid.*" By A. W. CROSSLEY and H. R. LE SUEUR.

The high boiling fraction of the liquid obtained by the action of phosphorus pentachloride on dimethyldihydroresorcin consists of 3:5-dichloro-o-xylene,  $C_6(H_2Cl)_2$ , which may also be produced by the prolonged heating of dichlorodimethyldihydrobenzene with phosphorus pentachloride. It is a faintly yellow, highly refractive liquid, which boils at 129° (23 mm.), or at 226° (atmospheric pressure), and on cooling solidifies to a mass of flaky needles melting at 3–4°.

On oxidation with nitric acid, it is converted into 3:5-dichloro-o-phthalic acid,  $C_6H_2Cl_2(CO_2H)_2$ , which crystallises in hair-like needles melting at 164° with previous contraction and sublimation. The anhydride (m. p. 89°) and several other derivatives have been prepared.

\*148. "*The Combination of Carbon Monoxide with Chlorine under the Influence of Light.*" By G. DYSON and A. HARDEN.

Following the method previously described (*Proc.*, 1894, x., 165), the authors have found:—

1. That when a mixture of carbon monoxide and chlorine, dried by being passed through sulphuric acid, is exposed to light, a well-marked period of photochemical induction occurs.

2. The induction lapses slowly when the exposed gas is placed in the dark.

3. The sensitiveness of this mixture to light is greatly diminished by admixture of air, but is not much affected by the presence of carbonyl chloride, hydrogen chloride, excess of carbon monoxide, vapour of carbon tetrachloride, and small amounts of water vapour.

#### DISCUSSION.

Professor TILDEN said one point which Dr. Harden had omitted to mention was the extent to which the gases had been deprived of moisture. He would be glad to hear by what method they had been dried, and whether the authors had observed that the composition of the glass

or the condition of the surface in contact with the gases produced any influence on the rate of their combination.

Dr. HARDEN replied that the gases were dried by being bubbled through strong sulphuric acid. They had not made any experiments specially with reference to the composition of the glass or the condition of its surface.

\*149. "The Constituents of Commercial Chrysarobin." By H. A. D. JOWETT, D.Sc., and C. E. POTTER, B.Sc.

Araroba, or Goa powder, and commercial chrysarobin, the substance obtained from the former by extraction with solvents as chloroform, have been investigated by Attfield, by Liebermann and Seidler, and by Hesse with varying results. The latter chemist found that crude chrysarobin contained no chrysophanic acid, but was a mixture of two parts of chrysarobin,  $C_{15}H_{12}O_3$ , and one of its methyl ether. He regarded chrysarobin as the anthranole of chrysophanic acid. The authors have examined commercial chrysarobin, and find that it contains the following substances, but no chrysophanic acid.

Chrysarobin,  $C_{15}H_{12}O_3$ , is the anthranole of chrysophanic acid and identical with chrysophanoanthrone obtained by the reduction of chrysophanic acid. It melts at  $204^\circ$ . When acetylated with acetic anhydride alone, a mixture of diacetylchrysarobin (m. p.  $193^\circ$ ) and triacetylchrysarobin (m. p.  $238^\circ$ ) is obtained, but if sodium acetate and acetic anhydride are used, the triacetyl compound is alone produced.

Methyl ether of dichrysarobin,  $C_{32}H_{26}O_7$ , melts at  $160^\circ$ . It yields a soluble pentacetyl compound, m. p.  $135^\circ$ , identical with Hesse's hexacetyldichrysarobin.

Dichrysarobin,  $C_{30}H_{24}O_7$ , does not melt below  $250^\circ$ , but blackens and chars gradually. On acetylation, hexacetyldichrysarobin (m. p.  $179-181^\circ$ ) is obtained. The formula assigned to this constituent is proved by analysis.

A substance,  $C_{17}H_{14}O_4$ , m. p.  $181^\circ$ , which yields an acetyl compound, m. p.  $215-216^\circ$ .

The two latter substances are contained in crude chrysarobin in only small amount, the greater portion consisting of the two first named substances. Methylchrysarobin was not found to be a constituent of crude chrysarobin. Both chrysarobin and dichrysarobin yield chrysophanic acid on oxidation, and  $\beta$ -methylanthracene by distillation with zinc dust.

Crude chrysophanic acid as obtained from rhubarb contains pure chrysophanic acid, m. p.  $190^\circ$  (acetyl compound, m. p.  $206^\circ$ ), and the methyl ether of dichrysarobin, and not the methyl ether of chrysophanic acid as previously stated by Hesse.

\*150. "The Constituents of an Essential Oil of Rue." By F. B. POWER and F. H. LEES.

In order to prepare some methyl nonylketone, the authors obtained a quantity of oil of rue labelled *Ol. Ruta. Ang.*, from an English distiller, and found subsequently, on inquiry, that it was not distilled from the native-grown herb. From their results, taken in conjunction with those of Thoms (*Ber. Deut. Pharm. Ges.*, 1901, x., 3), and von Soden and Hexle (*Pharm. Zeit.*, 1901, xlv., 277 and 1026), the authors regard it as most probably of Algerian origin.

The oil was light brown in colour.  $d_{15.5/16^\circ} = 0.8405$ ,  $n_D = -3^\circ 48'$  for a 100 m.m. tube. It was completely soluble in two parts of 70 per cent alcohol. The following constituents were identified:—(1) Methyl *n*-heptylketone, b. p.  $194.5-195.5$  at 763 m.m.,  $d_{14/16^\circ} = 0.8296$  (semicarbazone, m. p.  $119-120^\circ$ ); (2) methyl *n*-nonylketone, b. p.  $231.5-232.5^\circ$ ,  $d_{20.5/16^\circ} = 0.8263$  (semicarbazone, m. p.  $122^\circ$ ); (3) methyl *n*-heptylcarbinol, b. p.  $198-200^\circ$ ,  $d_{19/16^\circ} = 0.8273$ ,  $n_D = -3^\circ 44'$  in a 50 m.m. tube; acetate, b. p.  $213-215^\circ$ ,  $d_{20.5/16^\circ} = 0.8605$ ,  $n_D = -3^\circ 3'$  in a 50 m.m. tube (oxidation of alcohol to the corresponding ketone, b. p.  $195-199^\circ$ , semicarbazone, m. p.  $118-119^\circ$ ); (4) methyl *n*-nonylcarbinol, b. p.  $231-233^\circ$ ,  $n_D = -1^\circ 18'$  in a 25 m.m. tube (oxidation to corresponding ketone, oxime, m. p.  $46-47^\circ$ ); (5) a blue oil of high but not constant boiling-point; (6) acetic acid; (7) a basic

substance having the odour of quinoline; (8) a mixture of free fatty acids; (9) methyl salicylate; (10) an ester of valerianic acid, apparently ethyl valerianate; (11) pinene (nitrosochloride, nitropiperidine, m. p.  $119-120^\circ$ ; (12) *l*-limonene (tetrabromide, m. p.  $103^\circ$ ); (13) cineol (compound with tetraiodopyrrol, m. p.  $114-115^\circ$ , and hydrobromide, m. p.  $55-56^\circ$ ).

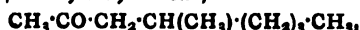
The relative proportions in which the above-mentioned substances were found to exist in the oil are approximately as follows:—The two ketones constitute 80 per cent, and were present in about equal amounts. The two alcohols represent about 10 per cent, the methyl *n*-heptylcarbinol preponderating; they were present partly in the free state and partly as acetic esters. The terpenes, together with cineol, amount to about 1 per cent, and the blue oil to about 0.5 per cent. From the non-ketonic portion of the oil, a small amount of an undistillable, viscous substance, probably a decomposition product, was separated.

Since the substances associated with the ketones and alcohols are present in such small amount, and the limonene is the more rarely occurring *l*-form, the authors conclude that all these substances are natural constituents of the oil.

\*151. "Methyl  $\beta$ -Methylhexyl Ketone." By F. H. LEES.

Ethyl *sec*-hexylacetoacetate,  $CH_3 \cdot CO \cdot CH(C_6H_{13}) \cdot CO_2Et$ , was prepared by the condensation in alcohol at  $100^\circ$  of ethyl sodioacetoacetate with *sec*-hexyl iodide; it is a colourless oil which boils at  $130-132^\circ$  (17 m.m.), and at  $243-245^\circ$  under atmospheric pressure.

Methyl  $\beta$ -methylhexyl ketone,—



was prepared by the hydrolysis of ethyl *sec*-hexylacetoacetate with 30 per cent aqueous potassium hydroxide. It is a colourless oil with a pleasant, fruity odour, boiling at  $184^\circ$  (769 m.m.), and having the density  $d_{15/16^\circ} = 0.8319$ . Its semicarbazone forms needles and melts at  $75^\circ$ ; its oxime is a colourless oil.

(To be continued).

## PHYSICAL SOCIETY.

Ordinary Meeting, November 14th, 1902.

Prof. S. P. THOMPSON, President, in the Chair.

A PAPER ON "The Theory of the Aluminium Anode," by W. W. TAYLOR and J. K. H. INGLIS, was read by Mr. INGLIS.

Aluminium is very slowly acted upon by dilute sulphuric acid even at moderately high temperatures. With dilute hydrochloric acid the action is violent, and it is found that if a little hydrochloric acid or soluble chloride be added to dilute sulphuric acid, the action is as violent as with hydrochloric acid of the same concentration. The object of the present paper is to find an explanation of this anomalous behaviour of sulphuric acid, and of the effect produced by the addition of chloride. It has long been known that, when an aluminium electrode is employed as anode in a solution of a sulphate or sulphuric acid, there is a very great resistance offered to the current, and that this resistance is due to a film which separates the electrode from the solution. If the aluminium is the cathode, or if other acids are substituted for sulphuric acid, this great resistance does not exist. It seems probable that the two phenomena are related, and that the film is also the cause of the slow action of sulphuric acid on aluminium. The authors have attempted to establish a theory which will explain these phenomena. The paper opens with a historical account of the subject; and it is pointed out that the question is now of practical importance in con-



nection with the use of aluminium electrodes for the conversion of alternating currents into direct currents. The first experiments were made with a cell consisting of aluminium and platinum electrodes in dilute sulphuric acid, and were carried out to ascertain in what way the addition of certain salts affected the aluminium anode. The influence of potassium chloride, bromide, nitrate, acetate, chlorate, and thiocyanate in various concentrations was investigated, and the authors conclude that the presence of certain ions enables a large current to pass through the cell. The reason seems to be that the film of aluminium hydroxide with which the anode is covered is permeable to certain ions but impermeable to others. The anomalous behaviour in sulphuric acid would then be due to the impermeability of the film to the  $\text{SO}_4^{--}$  ions, and also to the  $\text{Al}^{+++}$  ions. This explanation is in accord with the fact that reversal of the potential-difference acting upon the cell immediately causes a current to pass, the film being permeable to  $\text{H}^+$  ions. Experiments were next made upon the rates of diffusion of various ions through a film of aluminium hydroxide. It was found that several salts of potassium diffused rapidly through such a film, but that the sulphate passed through very slowly. This strengthens the argument that the abnormal behaviour of aluminium anodes in sulphuric acid is due to impermeability. It is suggested that the very small current which does pass may be due to a secondary action at the anode, in which the ions of water take part. The theory is confirmed by a series of experiments in which the peculiarities of the aluminium anode are reproduced by means of a platinum anode coated with a film of aluminium hydroxide. A further test was applied in the direct measurement of the resistance of a film of aluminium hydroxide to the passage of different ions. Quantitative experiments which the authors have performed on the effect of the presence of different ions on the reaction between aluminium and sulphuric acid, show that this reaction is considerably accelerated by the addition of potassium chloride.

Dr. LEHFELDT said that the lines of explanation were probably correct. Referring to the behaviour of an aluminium anode in sulphuric acid, he said that the small amount of conduction might be due to the fact that the film was not quite impermeable.

The CHAIRMAN called attention to one of the experiments described in the paper, in which a potential-difference of 50 volts applied to a cell with a membrane of aluminium hydroxide between the electrodes produced a current of 0.07 ampère, and asked what the current would have been if the voltage had been reversed.

Mr. CAMPBELL mentioned some experiments which he had seen performed with an aluminium rectifier. He said that if an alternating E.M.F. of small magnitude was applied to the electrodes of a cell consisting of two aluminium plates immersed in strong sulphuric acid, the electrostatic capacity of the cell was large, but if the E.M.F. was increased the capacity fell considerably, and then did not recover on reducing the applied E.M.F. till the cell had been allowed to rest for some considerable time. He asked for an explanation of this phenomenon.

Prof. AYRTON asked the authors whether the rectifying power of an aluminium anode was a question of resistance or of back E.M.F. He said that if an aluminium anode and a lead cathode were placed in a solution of Rochelle salt and an E.M.F. applied for about an hour, it was then impossible to send a current through the cell even by increasing the E.M.F. to 120 volts. If such a cell was used as a rectifier, a black deposit was formed on the aluminium plate which remained constant in weight. If a direct current was sent through the cell, a white deposit was formed and the aluminium plate lost in weight. Prof. Ayrton described some experiments which seemed to show that the properties of these cells were connected with changes in the liquids of the cells.

Mr. INGLIS, in reply to the Chairman, said that in the particular experiment referred to a reversal of the E.M.F. would have given a current of about 30 amperes. In the experiment mentioned by Mr. Campbell the explanation might be that when the E.M.F. was large the ions could permeate into the interstices of the film, and produce an effect corresponding to a diminution of electrostatic capacity. He thought that the power of an aluminium anode as a rectifier was due to increased resistance and not to back E.M.F.

A paper on "*A Determination of the Ratio of the Specific Heats at Constant Pressure and at Constant Volume for Air and Steam*" was read by Mr. MACKOWER.

The method employed in this paper is similar to that used by Lummer and Pringsheim, and consists in allowing the gas under investigation to expand adiabatically, and measuring the lowering of temperature caused by such expansion. The chief modification introduced was the substitution of a platinum thermometer with compensating leads for a bolometer strip. The electrical contacts were made by a specially constructed mercury switch, instead of by hand, and it was found possible to work with smaller quantities of gas than Lummer and Pringsheim had used. In the experiments with air the vessel used was about 50 litres capacity. The pressures were measured with an oil manometer, and the temperatures with the platinum thermometer. The time which elapsed between the instant of reducing the pressure and the reading of the final temperature was determined by means of a chronograph, and varied in the experiments described in the paper from 0.5 second to five seconds. The value of  $\gamma$  must be corrected on account of conduction and convection during this interval. A correction was also applied to eliminate the effect of radiation from the vessel to the thermometer. The author's value for the ratio of the two specific heats in the case of air is 1.401. In the case of steam, a cylindrical copper vessel of about 9.3 litres capacity was used. The vessel was enclosed in a copper jacket filled with steam so that the steam in the inner vessel was superheated about 10° C. The pressure in the jacket was kept constant by means of an automatic gas-regulator, devised by Prof. Callendar, which controlled the supply of coal-gas reaching the burner employed for the generation of the steam. The observations with steam were similar to those in the preceding experiments, but special precautions were necessary to prevent the condensation of the steam in the tubes leading to the vessel. The results for steam were not sufficiently accurate to justify the application of corrections for radiation, and for conduction and convection. The values of  $\gamma$  deduced from two series of experiments were 1.307 and 1.304.

Prof. CALLENDAR congratulated the author upon the good agreement of the results of a difficult experiment. He mentioned the difficulties in overcoming conduction when working with steam at a high temperature, and pointed out that the author's value for the ratio between the specific heats of steam agreed closely with that which he (Prof. Callendar) had recently deduced from theoretical considerations. An important feature of the present paper was that experiments had been performed in which different periods of time elapsed between the releasing of the pressure and the determination of the final temperature. Referring to the fact that the author had used a platinum thermometer instead of a bolometer strip, he said that although a bolometer strip was more sensitive than a platinum thermometer it was more sensitive than necessary in such an experiment. Another point in the experiments was the use of smaller quantities of gas than had hitherto been employed, an advantage of importance when dealing with rare gases. Prof. Callendar then exhibited and described the pressure-gauge referred to by the author and used in his experiments.

The Society then adjourned until November 28th.

ROYAL SOCIETY OF NEW SOUTH WALES.

THE General Monthly Meeting of the Society was held at the Society's House, No. 5, Elizabeth Street North, on Wednesday evening, October 8, 1902; Prof. WARREN, M.Inst.C.E., Wh.Sc., President, in the Chair.

The following papers were read:—

"Occurrence of the Mineral Gadolinite at Cooglegong, Pilbarra District, West Australia." By BERNARD F. DAVIS, B.Sc. (Communicated by Prof. DAVID, B.A., F.R.S.).

The specimen of gadolinite described in this paper was identified as such by Professor David and Mr. W. G. Woolnough, when it was brought over last year to Sydney University by Mr. Bernard F. Davis. Mr. Davis kindly undertook to make an analysis of the mineral, and place the results at the disposal of the Royal Society of New South Wales. The mineral occurs in lodes, associated with tinestone and monazite contained in gneiss. It was found by Mr. Davis at Cooglegong, in the Pilbarra District, West Australia. The following is the analysis by Mr. Davis:—

|                                                      |    |    |    |       |
|------------------------------------------------------|----|----|----|-------|
| SiO <sub>2</sub>                                     | .. | .. | .. | 23.33 |
| FeO                                                  | .. | .. | .. | 10.38 |
| BeO                                                  | .. | .. | .. | 12.28 |
| Ca <sub>2</sub> O <sub>3</sub>                       | .. | .. | .. | 2.50  |
| La <sub>2</sub> O <sub>3</sub> and Di <sub>2</sub> O | .. | .. | .. | 18.30 |
| Y <sub>2</sub> O <sub>3</sub>                        | .. | .. | .. | 33.40 |
| MgO                                                  | .. | .. | .. | 0.69  |
| Loss on ignition                                     | .. | .. | .. | 0.32  |

101.20

Dr. Norman Collie examined the mineral for helium and other gases. He says that 10 grms. on heating gave about 10 c.c. of CO<sub>2</sub> and 10 c.c. of hydrogen, a little nitrogen, and about one bubble of helium.

"Pot Experiments to Determine the Limits of Endurance of different Farm Crops for certain Injurious Substances. Part I. Wheat." By F. B. GUTHRIE and R. HELMS.

The authors describe experiments to test the effect upon the growth of the wheat plant of the following substances occasionally found in the soil and in manures, and which are known when present in excessive quantities to act as plant poisons:—Sodium chloride and sodium carbonate, which are present in many of the artesian bore-waters used for irrigating; ammonium sulphocyanide, which has been found in impure samples of ammonium sulphate; sodium chlorate, occasionally present in sodium nitrate; and arsenious oxide, which may be present in superphosphate which has been manufactured with acid derived from sulphur or pyrites containing arsenic. The experiments were carried out in cylindrical galvanised iron culture pots of familiar construction, and were located, with the kind permission of Mr. J. H. Maiden, in the Botanic Gardens. Details of the soil and manures employed, and of the appearance at different stages of the plants treated with varying proportions of the substances under examination were supplied. The following table summarises the principal results obtained:—

Effect upon Germination and subsequent Growth of Wheat of different Percentages of Injurious Substance in the Soil.

|                                   | Germination affected. | Germination prevented. | Growth affected.         | Growth prevented. |
|-----------------------------------|-----------------------|------------------------|--------------------------|-------------------|
| NaCl ..                           | 0.05                  | 0.20                   | 0.05 to 0.15 (recovered) | 0.20              |
| N <sub>2</sub> CO <sub>3</sub> .. | 0.30                  | 0.5 to 1.0             | 0.10                     | 0.40              |
| NH <sub>4</sub> CNS..             | 0.005                 | 0.01                   | 0.001                    | 0.005             |
| NaClO <sub>3</sub> .. Above       | 0.01                  | 0.05                   | 0.001                    | 0.003             |
| As <sub>2</sub> O <sub>3</sub> .. | 0.05                  | 0.50                   | 0.05                     | 0.10              |

OBITUARY.

SIR WILLIAM ROBERTS-AUSTEN, K.C.B., F.R.S.

It is with very great regret that we announce the death of Sir William Chandler Roberts-Austen, which occurred at the Royal Mint on Saturday last, November 22nd.

Sir William Roberts-Austen was born in 1843, and was therefore fifty-nine years of age. He was of Welsh descent through his father, whose name was George Roberts, and his mother belonged to the old Kentish family of Chandler. In 1885, at the request of his uncle, the late Major Austen, J.P., of Haffenden and Cambourne in Kent, Sir William (then Mr.) Roberts obtained Royal licence to take the name of Austen.

In 1861 he entered the Royal School of Mines with the intention of becoming a mining engineer, but on obtaining the Associateship, the late Professor Graham, then Master of the Mint, secured his services. With him he carried on a long series of researches, and on Professor Graham's death in 1869, Mr. Roberts succeeded to one of the appointments that he held, viz., that of Assayer to the Mint. Thirteen years later, in 1882, he became Queen's Assay Master.

In 1880, on the retirement of the late Dr. Percy, F.R.S., he was appointed to the Chair of Metallurgy at the Royal School of Mines, where less than twenty years previously he had been a student. This appointment he held till his death, though only a few weeks ago he was compelled, through failing health, to request his great friend, Dr. Gowland, to finish the current course of lectures on his behalf.

The present writer was one of those students who attended Sir William Roberts-Austen's first course of lectures in 1880, and he remembers well the enthusiasm and energy with which the late professor entered on his work. As a teacher he was a universal favourite, and was never tired of helping and encouraging his students, both past and present. It was Sir W. Roberts-Austen who inaugurated the annual tours for his students through some metallurgical district; the first one in 1881 to Swansea and the neighbourhood is still vivid in the writer's mind; just as the train was leaving Paddington the news was received of the death of Lord Beaconsfield.

Apart from his work as Chemist to the Mint, and his Professorship at South Kensington, Sir William Roberts-Austen was well-known and esteemed in purely scientific circles. As far back as the year 1868 he wrote a paper on "The Occurrence of Organic Appearances in Colloid Silica obtained by Dialysis," which was published in the *Journal of the Chemical Society*. In the following year he published "Some Experiments illustrating the Expansion of Palladium during the Formation of its Alloy with Hydrogenium." His principal work, however, was on alloys and metallurgical subjects, with which his name will always be closely connected; his last paper, in conjunction with Dr. T. K. Rose, published in the *CHEMICAL NEWS* in 1900 (vol. lxxxi, p. 241), being on "Certain Properties of Alloys of Gold and Copper."

Sir William was elected a Fellow of the Royal Society in 1875, and has held numerous posts, all of which he filled with distinction. He was President of the Iron and Steel Institute in 1899, Hon. General Secretary to the British Association for the Advancement of Science, Vice-President of the Chemical Society, the Physical Society—of which he was also one of the founders—and of the Society of Arts. He served on the British Executive of the Paris Exhibition of 1889, and was Vice-President of the International Mining and Metallurgical Congress in Paris, receiving from the President of the Republic the Cross of Chevalier of the Legion of Honour.

In 1888 he was made a C.B., and in 1899 he was created K.C.B. He was also a D.C.L. of the University of Durham.

Among other appointments, Sir William was a member

of the Explosives Committee of the War Office, where his knowledge and experience in metallurgical matters made his opinion of great value.

Though in failing health, his death was unexpected, and he will be sincerely mourned by a large number of friends, scientific and otherwise.

## NOTICES OF BOOKS.

*Chemisches Praktikum. I. Teil. Analytische Übungen* ("Practical Chemistry. Part I. Analytical Exercises"). By Dr. A. WOLFRUM. Leipzig: Wilhelm Engelmann. 1902.

THIS course of practical chemistry comprises both elementary and advanced technical analysis. The book is divided into three parts. The first deals with qualitative analysis of inorganic and organic substances, including under the latter heading many technical products; this section being particularly well written. Some parts of the introduction, however, might be revised with advantage; for instance, the discussion of the degree of dissociation on page 5 is distinctly misleading. The methods of detection of organic substances will certainly be found useful, being concise and in a handy form for reference. The second part of the book is devoted to quantitative analysis in all its branches, including all ordinary methods of determination. More detailed descriptions of some of the experiments would improve the book, regarded from the point of view of the student who relies on it alone for his information. The last part treats of quantitative technical analysis of various commercial products, but no food products are included. As far as possible, having regard to the necessarily restricted space devoted to this section, the accounts appear to be very complete.

*Beiträge zur Chemischen Physiologie und Pathologie.* ("Contributions to Chemical Physiology and Pathology"). Edited by FRANZ HOFMEISTER. Band III. 1-3 Heft. Braunschweig: Friedrich Vieweg und Sohn. 1902.

THIS recently issued number of the *Beiträge* begins the third volume. The six papers which it contains fully maintain the high standard of interest of former numbers. They include a paper by E. Friedmann describing researches aimed at determining the constitution of cystin, and enumerating the properties of some of its derivatives and another on the action of chloroform on hæmoglobin; the evidence seems to point to the fact that this action, brings about essential changes in the structure of the hæmoglobin molecule. A third paper, by Fr. N. Schulz and R. Zeigmondy, gives the result of investigations of the "gold number" of various albuminous substances. Apparently this number, of which the definition according to Zeigmondy is given, may, like other physical and chemical properties, such as optical activity and temperature of coagulation, be regarded as a definite means of distinguishing albumens.

*Merck's Index.* Second Edition. Darmstadt: Eduard Roether. 1902. Pp. 374.

THE first edition of this book, published five years ago, being exhausted, it has been found advisable to issue a second. The same plan has been adhered to as was adopted for the first edition, but its scope naturally has been much enlarged, and a good deal of attention has been devoted to the needs of physicians and pharmacists.

The name of "Merck" in connection with this subject is very widely known, and the firm is constantly bringing forward new drugs derived from the never-failing coal-tar

products; we must, however, urge that great caution be exercised in their use, especially at first. What is one man's meat is another man's poison, and the same can be truly applied to many of these new preparations. Prolonged and careful experiments are necessary before many of these bodies should enter into general use.

The book is divided into six parts. The first comprises chemical bodies and definite principles, certain galenic preparations and organic extracts; the second, special solutions and forms of substances used in analytical and microscopical work; the third, crude drugs; the fourth, minerals; the fifth, collections suitable for schools and museums; and the sixth, sundries.

A noticeable feature in this new edition is the information on the chemical and physical properties of the substances included, and also that on the technical application of various bodies.

*The Painters' Laboratory Guide, and Handbook on Paints, Colours, and Varnishes for Students.* By GEORGE H. HURST, F.C.S., &c. With 22 Illustrations. London: Charles Griffin and Co., Ltd. 1902. Pp. 248.

IF the author had called this book the "Paint Makers'" laboratory guide he would have expressed the character of the work better than is done by the present title. In his opening sentences he shows that it is with paints on the large scale that we have to deal, and these paints may be described as fulfilling two objects, viz., protection and decoration.

The book is divided into ten chapters, of which five (Chapters II. to VI.) deal with the preparation of pigment colours. As a general rule the author first describes the pigment, whether a natural or artificial product; if the former, the localities whence it is obtained are mentioned; if the latter, the processes of manufacture are described, with instructions for carrying them out on a small scale in the laboratory when possible. Chapter VII. is on "Lakes," and here the author has given special attention to their preparation from coal-tar dyes owing to the rapidly increasing importance of these bodies. The class of pigments known by the name of lakes is possessed of special properties that serve to distinguish them from purely mineral pigments; they owe their colour to organic colouring matters and dye-stuffs, and in more recent years the use of the coal-tar dyes has been pressed into service to replace the natural products, such as cochineal, logwood, &c.; still it is essential that a metallic oxide be present in their preparation, and it is this oxide entering into combination with the acid principle of the organic matter used that forms the basis of lake-making. In this section the author has introduced a simplification in the manner of writing the names of the coal-tar derivatives; instead of running a dozen syllables or more all together, as is unfortunately the present custom; he sub-divides the word into its natural components by means of hyphens, thus:—Para-nitro-benzene-azo-beta-naphthol instead of par-nitrobenzeneazobetanaphthol; there can be no question as to which is the more easily read and quickly understood, and though we are afraid it is asking a great deal, we should be very glad to see the change made generally.

There is no legitimate reason why what is really a short descriptive sentence should be printed and written as if it were simply one word, and when sub-divided, as the author has done in this volume, the names of the organic products are both easier and quicker to read. So much work in organic chemistry has been done in Germany, that chemists have unfortunately adopted the German style of combining all the descriptive adjectives into one word, "portmanteau" words Mark Twain called them; but the method is clumsy and quite as un-English as if we were to write down a certain steam-engine as a fifteenhousandhorsepowerreciprocatingtripleexpansion-condensingmarinesteamengine, or to write of extra

superfinecream laid blue ruled white foolscap writing paper, for example.

Chapters VIII., IX., and X. are on "Paint-oils and Thinners," "Driers," and "Varnishes," respectively, and form an important section, while the book closes with a good index of seven pages of double columns.

## CORRESPONDENCE.

### "FIXING" NITROGEN FROM THE ATMOSPHERE.

To the Editor of the Chemical News.

SIR,—The combination of atmospheric oxygen and nitrogen by electric arc, with formation of nitric acid or nitrate, as sketched in the CHEMICAL NEWS (lxxxvi., p. 238) and elsewhere, will have the interesting consequences of diminishing the total weight of the atmosphere as well as of impoverishing the remaining atmosphere in oxygen. Oxides of nitrogen are formed, absorbed by water or alkali to nitrates and nitrites. That is,  $\text{NO}_2$  is formed (subsequent conversion to all nitrate by atmospheric oxygen would mean  $\text{N}_2:\text{O}_2$ ), containing about 70 per cent by weight of oxygen, from air containing about 23 per cent by weight of oxygen.

In all fuel and other combustion the oxygen of the atmosphere is diminished; in this combustion of atmospheric nitrogen, the nitrogen as well as the oxygen is for the time being taken out of the atmosphere, and in undesirable ratio.

The weight of remaining atmosphere will be very large indeed, but an annual 12 million tons of nitrate of soda would be a serious additional draft on the oxygen of the atmosphere. The increase of green leaf surface would not be more than proportional to increase of population.—I am, &c.,

W. H. DERRING.

Woolwich, November 17, 1902.

### ANALYSIS OF APATITE.

To the Editor of the Chemical News.

SIR,—The following is the analysis of some crystals of apatite that were obtained near Antwerp, Jefferson County, New York, some time ago. They were olive-green prisms, and were about 2 c.m. long and 8 m.m. in diameter. The analysis was made in the Chemical Laboratory of Cornell College by Assistant Frank L. Hann.

|                                      | Per cent. |
|--------------------------------------|-----------|
| $\text{P}_2\text{O}_5$ .. .. .       | 41.0      |
| $\text{CaO}$ .. .. .                 | 48.2      |
| $\text{Ca}_3(\text{PO}_4)_2$ .. .. . | 89.2      |
| $\text{SiO}_2$ .. .. .               | 0.6       |
| $\text{Al}_2\text{O}_3$ .. .. .      | 9.0       |
| $\text{CaF}_2$ .. .. .               | 1.2       |
|                                      | 100.0     |

—I am, &c.,

NICHOLAS KNIGHT.

Mount Vernon, Iowa,  
November 4, 1902.

Action of Isocyanate of Phenyl on the Ethers of some Oxy-acids.—E. Lambling.—In a long paper the author describes a number of experiments on the action of isocyanate of phenyl on the ethers of glycolic, lactic, and phenylglycolic acids, and he describes, for each of these acids, the phenylurethane of the ether, that of the corresponding acid and the lactame derived from this acid.—*Bull. de la Soc. Chim. de Paris*, xxvii., No. 10.

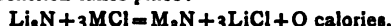
## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxv., No. 18, November 3, 1902.

Aluminium Chlorosulphate.—A. Recoura.—By crystallisation of a solution of aluminium sulphate in boiling hydrochloric acid the author obtains the chlorosulphate of aluminium,  $\text{AlSO}_4\text{Cl}_6\text{H}_2\text{O}$ . This substance is very soluble in water and very slightly soluble in alcohol. Cryoscopic measurements show that the solution in water is, not even at first, anything more than a simple mixture of aluminium sulphate and chloride. The lowering of the freezing-point of the aqueous solution of this compound is the sum of the respective lowerings of the sulphate and the chloride. The various experiments performed on this salt lead the author to believe that it has the same constitution as chromium chlorosulphate.

A General Method of Preparing Metallic Nitrides.—M. Gunz.—The author in a previous research measured the heat of formation of lithium nitride. If this substance is allowed to react on a metallic chloride,  $\text{MCl}$ , the following reaction takes place:—



and the number  $Q$  is generally very considerable, because lithium chloride is one of the chlorides formed with the largest evolution of heat. Thus, in the following reaction if  $x$  is the heat of formation of the ferrous nitride—

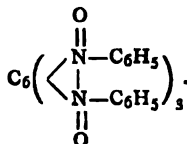


There is a considerable excess of heat in this case, allowing for the formation of the ferrous nitride. The author, therefore, uses such a reaction to prepare the nitrides of iron,  $\text{Fe}_2\text{N}_2$  and  $\text{FeN}$ .  $\text{Li}_3\text{N}$  acts on  $\text{FeCl}_2\text{KCl}$  in a very energetic manner. The two products should first be pulverised, and then gently mixed in a crucible. This is heated, and after cooling the contents washed with boiling water and then dried at  $100^\circ$ . The nitride thus obtained,  $\text{FeN}$ , is a black compound which when heated on platinum foil becomes incandescent and is transformed into iron oxide.

Barium-ammonium and Barium-amide.—M. Mentrel.—It has already been shown that metallic barium and strontium dissolve in liquid ammonia to give definite compounds. The author now examines the conditions of formation of barium-ammonium and its properties. His method of preparation consists in passing ammonia gas over barium. The metal is not attacked above  $+28^\circ\text{C}$ . Below this temperature a red solid product is obtained which is transformed into a blue liquid when the temperature sinks below  $-23^\circ$ . Towards  $-50^\circ$  an oily dark blue liquid separates, slightly soluble in liquid ammonia, colouring it blue. Below  $-23^\circ$  these compounds are stable; at  $-15^\circ$  they are transformed into the amide, and this transformation takes place more rapidly as the temperature is raised. An analysis of the various products gives:—At  $0^\circ$ ,  $\text{Ba} + 6.1\text{NH}_3$ ; at  $-23^\circ$ ,  $\text{Ba} + 6.3\text{NH}_3$ ; and at  $-50^\circ$ ,  $\text{Ba} + 6.97\text{NH}_3$ . At low temperatures, therefore, the barium-ammonium contains ammonia in slight excess, proving the solution of this gas in the solid compound. The formula of the latter apparently is  $\text{Ba}(\text{NH}_3)_6$ .

Some Oxidation Products of Aniline.—C. I. Istrati.—The air has a considerable action on aniline at its boiling-point. At the same time as it unites with oxygen condensation takes place. During the reaction a quantity of water is produced, and a little ammonia. After ten hours of boiling, the aniline becomes brown; after ten days, it is brown and viscous. Gradually it deposits blackish crystals, and the mass becomes completely solidified by about the twenty-fifth day. This mass is filtered and washed with cold alcohol. A brownish red substance remains on the filter and a black liquid passes

through; this latter containing all the non-oxidised aniline. Chloroform dissolves out from the former substance a dark red material, having apparently the formula  $(C_6H_5-NH)_2 \equiv C_6H_2-O-C_6H_2 \equiv (NH-C_6H_5)_2$ . The portion insoluble in chloroform, on purification, yields long colourless crystals melting at  $238-239^\circ$ , and apparently of the formula—



**Estimation of Carbon Monoxide and Carbon Dioxide in Vitiated Air.**—Ferdinand Jean.—The author devises a simple and practical apparatus by which the amount of carbon monoxide and carbon dioxide in confined spaces may be rapidly estimated.

## MISCELLANEOUS.

**Phosphates of Rubidium and Cæsium.**—Ed. von Berg.—These substances are obtained by saturating  $PO_4H_3$  by the bases or their carbonates. *Monorubidic phosphate*,  $PO_4RbH_2$ , is obtained from its solution in beautiful quadratic prisms, by concentrating first by heat and then in the desiccator; it has an acid reaction, is very soluble in water, but insoluble in alcohol. *Dirubidic phosphate*,  $PO_4Rb_2H + H_2O$ , is very difficultly crystallisable in indistinct grains, very soluble in water, insoluble in alcohol, and has an alkaline reaction. This salt was obtained by leaving the product obtained by the addition of concentrated ammonia to its solution, in the desiccator over sulphuric acid; a slightly soluble white precipitate is formed of a double phosphate which gradually loses its ammonia in the desiccator. *Trirubidic phosphate*,  $PO_4Rb_3 + 4H_2O$ , occurs in badly formed prisms, and is very hygroscopic. *Metaphosphate of rubidium*,  $PO_3Rb$ , is obtained by the calcination of the monorubidic salt as a white powder, absolutely infusible, very soluble, and with a neutral reaction. *Pyrophosphate of rubidium*,  $P_2O_7Rb_4$ , is the result of the calcination of the dirubidic salt; it appears as a white fused mass, and has a neutral reaction. *Monocæsic phosphate*,  $PO_4CsH_2$ , in beautiful tabular crystals, is obtained in the same manner as the rubidium salt. *Dicæsic phosphate*,  $PO_4Cs_2H + H_2O$ , occurs in white microcrystalline masses, and is very soluble. *Tricæsic phosphate*,  $PO_4Cs_3 + 5H_2O$ , resembles the last salt. *Metaphosphate of cæsium*,  $PO_3Cs$ , is a white mass, coarsely granular, very difficult to fuse, soluble in water, and has a neutral reaction. *Pyrophosphate of cæsium*,  $P_2O_7Cs_4$ , forms a colourless glass; it is very deliquescent, and has an alkaline reaction.—*Berichte*, vol. xxxiv., p. 418r.

**The Acetylacetic Compounds of Platinum.**—A. Werner.—For the sake of convenience the term acetylacetonone will be represented in this note by the formula  $AcH$ . A boiling solution of chloroplatinate of potassium being treated with  $KOH$ , not in excess, and shaken up with  $AcH$ , soon becomes cloudy, and deposits orange coloured crystals; then after a few hours, further crystals of a bright yellow colour. The former are fairly soluble in water, but insoluble in the organic solvents, slightly soluble in water containing  $KOH$  or  $KCl$ , and give no colouration with  $Fe_2Cl_6$ . Their composition is  $PtClAc.KCl$ . The yellow salt is distinguished from the preceding one, in that its aqueous solution gives a yellow precipitate with hydrochloric acid, and this amorphous precipitate is only slightly soluble in water, but soluble in the organic solvents. The formula of the yellow soluble salt is  $PtAc_2.KCl$ , and the yellow precipitate formed by hydrochloric acid is the acid correspondent  $PtAc_2.HCl$ . The corresponding rubidium salt is soluble in water, and crystallises in alcohol in long yellow needles. A pre-

paration analogous to the one described above, but with an excess of potash, still gives a yellow crystalline precipitate, but of a more simple composition; this is a platinous acetylacetonate,  $PtAc_2$ . It can be crystallised in benzene in beautiful yellow crystals, and is not decomposed by strong acids. Finally, if we operate in an analogous manner on chloroplatinate of potassium with  $AcH$  and a strong solution of soda, we observe first that golden-yellow crystals are deposited, followed by a second lot of less soluble greenish yellow crystals. The first salt is soluble in water, and less soluble in the presence of alkalis; the solution is coloured deep red by  $Fe_2Cl_6$ , and gives precipitates with the metallic salts. This salt is  $PtAc_2.2NaCl + 5H_2O$ . If we boil its aqueous solution it changes to a brown colour; if we stop this action before it becomes black and add  $NaCl$ , we obtain a deposit formed of a yellow salt contaminated with resinous products. On taking up with chloroform, we obtain beautiful yellow crystals, very soluble in water; this salt is  $PtAc_2.NaCl + 2H_2O + CH_3Cl$ . The aqueous solution, treated with hydrochloric acid, gives a precipitate of  $PtAc_2.HCl$ .—*Berichte*, vol. xxxiv., p. 2584.

## MEETINGS FOR THE WEEK.

MONDAY, Dec. 1st.—Society of Arts, 8. (Cantor Lectures). "The Future of Coal-gas and Allied Illuminants," by Prof. Vivian B. Lewes.

Royal Institution, 5. General Monthly Meeting. Society of Chemical Industry, 8. "The Influence of Impurities on the Specific Gravity of Sulphuric Acid" and a "Note on the Determination of the Strength of Sulphuric Acid," by Arthur Marshall, F.I.C., F.C.S. "The Interaction of Sulphurous and Nitrous Acids as affecting various Absorbents Employed in Testing the Gases escaping from Vitriol Chambers," by R. Forbes Carpenter, F.I.C., &c., and J. E., Linder, B.Sc., &c.

WEDNESDAY, 3rd.—Society of Arts, 8. "Some Aspects of Photographic Development," by Alfred Watkins

THURSDAY, 4th.—Chemical, 8. "The Absorption-spectra of Metallic Nitrates" (Part II.), by W. N. Hartley. "The Specific Heats of Liquids," by H. Crompton. "Studies in the Camphane Series—Part X., The Constitution of Endolic Benzoylcamphor" and "Note on the isomeric Benzoyl Derivatives from Isonitrosocamphor," by M. O. Forster. "The Constitution of the Products of Nitration of Meta-acetolulide," by J. B. Cohen and H. D. Dakin.

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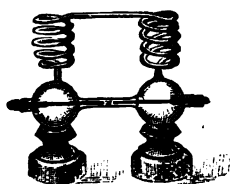
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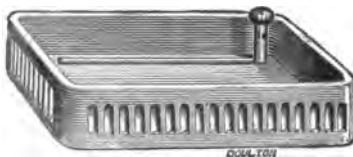
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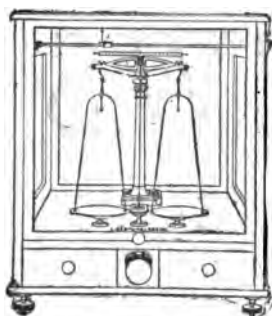
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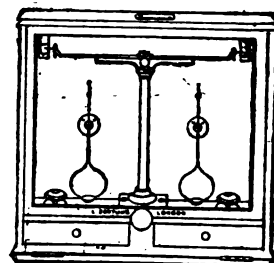


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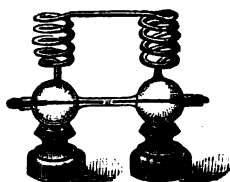
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# THE CHEMICAL NEWS.

VOL. LXXXVI., No. 2245.

## CRYSTALLISED PEROXIDE OF HYDROGEN.

By WILHELM STÄDEL.

THE firm of E. Merck, of Darmstadt, has been supplying a solution of peroxide of hydrogen at 30 per cent for some years past; this peroxide fulfils all our requirements as regards purity and stability. This preparation is appreciated as an antiseptic, more especially in connection with surgical instruments and appliances. They have been able to manufacture also a sufficiently concentrated solution on a large scale. The author of this article is indebted to the courtesy of this firm for large quantities of the latter product for the purposes of research.

This research is not yet completed, but it is possible, nevertheless, to announce one fact chosen from among the most interesting ones observed, viz., that peroxide of hydrogen, contrary to recent assertions, crystallises with ease, and in a very distinct manner. Its melting-point appears to be about  $-2^{\circ}$ , but it may be possibly a little higher. The preparation used contained from 95 to 96 per cent of  $H_2O_2$ . In a freezing mixture at  $-20^{\circ}$  it remained liquid; in a mixture of ether and solid carbonic anhydride it solidified into a hard resisting mass. Further, this phenomenon also occurred in chloride of methyl. The eutectic point thus appears to be between  $-20^{\circ}$  and  $-23^{\circ}$ . If we throw a trace of the solid peroxide into the liquid cooled down to  $-8^{\circ}$  or  $-10^{\circ}$ , there immediately form magnificent needle-shaped crystals, as transparent as water, and which soon permeate the whole mass. By draining the mother-liquor from the crystals and allowing these to regain their liquid state by fusion, we can obtain, by means of a second crystallisation, peroxide of hydrogen free from water.

Repeated analyses have given the composition of these crystals as 100 per cent  $H_2O_2$ . Similar crystals can be obtained from more dilute solutions titrating 90 or even 80 per cent of  $H_2O_2$ . It seems that practically we may look upon it as possible to prepare a pure peroxide of hydrogen without having recourse to an operation, which is not always free from danger; that is to say, to the distillation of high percentage solutions, as done by Wolfenstein and Brühl, or Spring, to obtain anhydrous peroxide of hydrogen.

For the better manifestation of the most marked properties of peroxide of hydrogen, a series of experiments was devised. Thus, an extremely small quantity of platinum black produced a catalytic decomposition accompanied by a violent explosion; powdered binoxide of manganese, or a mixture of carbon and powdered magnesium with a trace of binoxide of manganese, takes fire immediately when put in contact with anhydrous peroxide of hydrogen, or thrown into its solution at 90–95 per cent. Finely divided iron (reduced iron) alone is without action; the liquid simply mixes with the powder, but if we add now a little binoxide of manganese a flame is produced, and the iron burns, throwing off sparks. Powdered lead becomes enflamed in the same manner. A few drops of anhydrous peroxide of hydrogen thrown on to wool or a moist sponge immediately bursts into flame.

Monohydrated sulphuric acid can be mixed with anhydrous peroxide of hydrogen if it is done at a low temperature.

The refrigeration must be sufficient to counteract the evolution of heat which takes place. If the operation is not conducted under these conditions, oxygen very rich in ozone is given off. The examination of this reaction is not yet complete; it may be possible to produce persulphuric acid by these means.

We can also make use of peroxide of hydrogen in two reactions. The most interesting one, that is to say, that of which the best use can be made, takes place with sulphate of titanium. By its help we are able to detect 1 part of peroxide of hydrogen in solution in 1,800,000 parts of water. When the ratio is only 1 : 18,000 the reaction is manifested by the appearance of a deep yellow colouration, becoming bright yellow for 1 in 180,000, and remaining yellowish in a fairly thick layer with 1 part in 1,800,000. The action on ceric sulphate and ammonia is less sensitive; it reaches its limit of sensitiveness when the solution is diluted to 1 in 180,000. It must also be remarked that if the solutions in which we have produced the titanium reaction are still coloured after several days, this cannot be said of the cerium solutions, which in a very few days become completely colourless.

We know that peroxide of hydrogen is capable of forming crystallised compounds with metallic salts. M. Tanatar has described some of these bodies. The compound containing cadmium lends itself very well to the demonstration. On pouring a 90–95 per cent solution of peroxide of hydrogen into a concentrated solution of chloride of cadmium the mass becomes thickened by the formation of small, fine, white silky flakes. When collected and dried it has been found that they contained about 23 per cent of peroxide of hydrogen.

It may be concluded from the above that the problem of the preparation of anhydrous peroxide of hydrogen on the large scale has been solved. The economic results of this achievement will be known to us in the future. But from the point of view of facts already known, we may say with safety, that the use in surgery of an antiseptic product incapable of introducing foreign matters into wounds, or of producing anything besides water and oxygen by its decomposition, is made practicable by the present preparation; we are perfectly justified in hoping for great results in this direction.

In conclusion, we may remark that anhydrous peroxide of hydrogen, prepared by means of crystallisation, appears to be easily transported. A well-packed sample was placed on a truck and set on a journey of 50 or 60 kilometres, without undergoing any change. A second sample was treated in the same manner for three days without experiencing any injury.—*Zeitschrift für Angewandte Chemie*, 1902, p. 642.

## LABORATORY NOTES.

By HORACE JERVIS.

**Eliminating Ammonium Salts.**—Ammonium salts accumulated during the course of mineral analyses may be driven off (prior to the estimation of small amounts of magnesia say) either by direct vaporisation or by heating with nitric acid. The latter procedure is considered the more elegant; but the former need not be the spluttering, tedious, and generally unsatisfactory operation it is oft represented to be. It goes very smoothly if, after evaporating the liquid to crystallisation of the salts in the porcelain basin, the mass is scraped into a platinum dish and the transfer then cleanly completed by wiping with a piece of ashless filter-paper. A cover of ashless paper is then pressed on to the surface of the semi solid mass. This prevents spitting and also enables the moisture to be easily driven off if the mouth of the dish is inclined towards a hot muffle. The paper so charred remains intact nearly to the end.

**Self-hard Alloys.**—The exigencies of modern steel-works require analyses to be made so quickly that a stream of work of a stereotyped kind is passing continually through each operator's hands. Anything unusual interrupts the rate of progress and is considered more or less a nuisance. This unphilosophic attitude of mind is not commendable, but it can hardly be avoided; it is a

difficult matter to be an artist under such conditions as a respectable artisan would find intolerable. Being so long accustomed to see work disappear at express rate by a mere turn of the wheel, as it were, such an operator hardly knows what to do with a refractory sample. He concludes that it is insoluble or incompletely decomposed if the reagents used do not make short work of it while you wait, and so we are growing accustomed to unusual mixtures of acids which affect a speedy dissolution of the sample, but are not without disadvantages; for example—

The decomposition of the ferro-alloys used for making the more modern self-hard steels (which contain 10 to 12 per cent chromium and about three times as much tungsten) are vigorously attacked by a mixture of nitric and hydrofluoric acid, and the resulting solution can be used for the estimation of tungsten and (with limitations) chromium, phosphorus, and manganese; but it cannot be used for the estimation of sulphur or silicon, and platinum ware is essential. Such alloys are incompletely decomposed when digested for an hour or two with aqua-regia. But on prolonged digestion the dark coloured insoluble residue becomes quite yellow. As several days are generally available for the complete analysis the following scheme is practicable.

Pour 10 c.c. nitric acid (1.42) and 25 c.c. hydrochloric acid over 2½ grms. of the alloy which has been crushed to pass a 60 sieve, and allow to digest in a covered beaker at a temperature well below boiling point, so that the liquid may be hardly evaporated. In two or three days, or less if the operation is kept going through the night, the sample is decomposed and may be evaporated, &c., as usual, and the residue used for the determination of tungsten and silicon and the filtrate for sulphur, phosphorus, chromium, and manganese. For the estimation of the two latter elements, however, a sodium peroxide fusion, which, on extracting, leaves all the chromium in the filtrate and the manganese in the residue, is to be preferred.

The amount of impurity (ferric and chromic oxides) associated with the tungstic oxide may be about 1 per cent of its weight. This was so in an assay just made of a typical alloy; but two or three times this amount would probably be found, if the assay was evaporated and strongly baked, as is customary when sulphur and phosphorus is being estimated.

## LACTATES OF MERCURY.

By MARCEL GUERBET.

LACTATES of mercury have been investigated already by Engelhardt and Maddrel (*Lieb. Ann.*, 1847, vol. lxiii., p. 95) and by Brüning (*Ibid.*, 1857, vol. civ., p. 194).

The two former writers described a mercurous lactate insoluble in water, which corresponded to the formula  $(C_3H_5O_3)_2Hg_2 + 2H_2O$ , and a basic mercuric lactate, soluble in water, of which the formula should be  $(C_3H_5O_3)_2Hg.HgO$ .

Brüning, following the directions give by the preceding writers for the preparation of this latter salt, obtained, like them, a lactate soluble in water; he believed he had identified it as a mercurous salt, and gave it the formula  $(C_3H_5O_3)_2Hg_2$ .

In face of these contradictions, I took up the examination of the lactates of mercury, and I am about to prove that neither the basic mercuric lactate of Engelhardt and Maddrel, nor the mercurous lactate of Brüning are definite compounds; they are mixtures of Engelhardt and Maddrel's mercurous lactate and of the mercuric lactate which I have succeeded in isolating in a state of purity, and of which I will describe the preparation.

**Mercurous Lactate.**—Engelhardt and Maddrel obtained this body by mixing a very concentrated hot solution of lactate of soda with a saturated solution of mercurous

nitrate. It is deposited on cooling in the form of rose-coloured crystals.

The colour of this salt is due to an impurity, for I have sometimes succeeded in obtaining it completely white; I may add that when dried at the ordinary temperature it contains only one molecule of water of crystallisation, and agrees with the formula  $(C_3H_5O_3)_2Hg_2 + H_2O$ .

To prepare this body, I boiled some lactic acid diluted with ten times its volume of water; in this manner the anhydrides (dilaetic acid and laetide), which are always present in the concentrated acid, are destroyed (Wislicenus, *Lieb. Ann.*, vol. clix., p. 181).

On the other hand, we prepare some mercurous oxide by pouring a cold solution of mercurous nitrate into a cold solution of potash in excess, and rapidly washing the precipitate in the dark; we then add the amount of dilute lactic acid necessary for the solution of the mercuric oxide, which takes place immediately. The solution is then evaporated at the ordinary temperature over sulphuric acid.

The salt is deposited on the sides of the glass in crystalline crusts formed of short, colourless, prismatic needles.

The estimation of the mercury in the form of mercurous chloride gave 67.02 per cent of the salt used. The formula  $(C_3H_5O_3)_2Hg_2 + H_2O$  requires 67.11 per cent. Engelhardt and Maddrel found 64.86 per cent of mercury in their rose-coloured salt; it was estimated in the dry way, which, as we know, generally gives rather low results. They gave the salt the formula  $(C_3H_5O_3)_2Hg_2 + 2H_2O$ , which corresponds with 65.14 per cent of mercury.

The water cannot be estimated directly in this salt, as it is decomposed by heat.

Mercurous lactate is not completely soluble in water; it becomes disaggregated, one part dissolves, and the other part separates as a white powder which gradually becomes grey in appearance. If the mixture is heated it becomes black, and mercury is precipitated. The portion soluble in cold water consists principally of mercurous lactate with a little mercuric lactate. As the salt is completely soluble in water containing lactic acid, it is probable that the effect of water alone is, first to dissociate it into lactic acid, by which means a portion is dissolved, and into a more basic insoluble lactate; then there follows the decomposition of the insoluble portion into mercuric lactate and mercury, which gives it the grey appearance.

**Mercuric Lactate.**  $(C_3H_5O_3)_2Hg$ .—Engelhardt and Maddrel's basic mercuric lactate is not a definite compound, as I will show later on. I was able, however, to obtain a perfectly definite mercuric lactate in the following manner:—

Lactic acid diluted with ten times its volume of water, and boiled for half-an-hour to get rid of the anhydrides it contains, is treated with an excess of recently prepared yellow oxide of mercury. Solution takes place immediately, and it can be filtered at once. The solution is then evaporated over sulphuric acid at the ordinary temperature, and the salt gradually crystallises out. In spite of all precautions taken in the preparation of the yellow oxide, and in the saturation of the lactic acid, which should be effected without allowing any disengagement of heat, there is always a small quantity of mercurous lactate formed, which remains in the mother-liquors. To obtain a mercuric lactate entirely free from this latter substance, it is necessary to wash the crystals with a few drops of water. They are then dried at the ordinary temperature.

**Analysis.**—1.415 grms. of the salt dried over sulphuric acid, dissolved in water and reduced by phosphorous acid in the presence of hydrochloric acid, gave 0.880 gm. of mercurous chloride, which corresponds to 52.82 per cent of mercury. The formula  $(C_3H_5O_3)_2Hg$  requires 52.31 per cent.

**Properties.**—Mercuric lactate is deposited from its aqueous solutions in the form of colourless, prismatic needles grouped together in bundles.

It is very soluble in water; 1 part of water at 20° dissolves 2.75 parts of the salt. Under the influence of heat the solution undergoes a curious decomposition.

If it is boiled there is no appearance of any transformation, but in reality the mercuric salt gradually changes to the mercurous salt; for the solution which at first gave no precipitate on the addition of hydrochloric acid, gives a precipitate of mercurous chloride if the reagent is added after a few seconds' boiling.

At the same time, carbonic acid is formed; this can be recognised by means of lime-water, and also ordinary aldehyde, recognised by its odour. The latter can also be detected by distilling a few drops of the liquid, which reduces an ammoniacal solution of nitrate of silver, colours red fuchsin which has been decolourised by sulphurous acid, and precipitates acetate of phenylhydrazine.

Further, at the same time, lactic acid is set free. It is observed that the boiled solution, treated with a drop of potash, gives a precipitate of oxide of mercury, disappearing on agitation, while the original liquid leaves a permanent precipitate of this oxide on the addition of a trace of potash.

The transformation effected by boiling takes place according to the formula:—



Now that we know the properties of the lactates of mercury, let us return to the lactates prepared by Engelhardt and Maddrel, and by Brüning.

The first of these salts should have the formula  $(C_3H_5O_3)_2Hg.HgO$ . The authors obtained it by saturating a dilute boiling solution of lactic acid with red oxide of mercury, filtering, concentrating to a syrupy consistence, and allowing it to crystallise. First of all, yellow crystals insoluble in water were deposited, then very soluble, colourless crystals. On taking up with boiling water, filtering and evaporating over again, brilliant prismatic crystals were obtained, grouped round a centre; they were very efflorescent and easily soluble in both hot and cold water.

Brüning, following the above instructions, obtained, like Engelhardt and Maddrel, a salt soluble in water, and undecomposed by boiling water. Its solution, treated with hydrochloric acid, allowed the whole of the mercury present to be precipitated in the form of mercurous chloride; further, he gave it the formula of a mercurous lactate,  $(C_3H_5O_3)_2Hg_2$ .

These different chemists, working under the same conditions, thus obtained, the two former a *mercuric lactate*, the latter a *mercurous lactate*.

In face of this disagreement, I repeated their experiments, and following their instructions exactly, I vainly endeavoured to obtain a soluble lactate of mercury of a definite composition. I could only obtain various mixtures of Engelhardt and Maddrel's mercurous lactate, and the mercuric lactate of which I have already described the preparation. When these mixtures of the salts are rich in mercuric lactate they are soluble in water; on the other hand, they are only slightly soluble and dissociable when they are rich in mercurous lactate.

The following are the facts I have observed:—

When dilute lactic acid is boiled with red oxide of mercury, aldehyde is given off, as already observed by Brüning, and by passing the gas given off through lime-water we observe that carbonic acid is also evolved.

The solution, filtered and evaporated to a syrupy consistence, deposits the yellow crystals noticed by Engelhardt and Maddrel.

If we take up with boiling water, filter, and evaporate again, crystals are deposited which answer to the description given by these chemists, except concerning their solubility in water.

The crystals I obtained from this second crystallisation do not dissolve entirely in cold water; they leave a residue of a white powder, becoming grey and then black

when heated. Here we recognise the properties of mercurous lactate.

But these crystals also contain mercuric lactate, as their solution in water containing lactic acid and precipitated by hydrochloric acid, gives a fresh precipitate of mercurous chloride if we add phosphorous acid.

If we estimate the mercury, we find that the salt contains a minimum 65.07 per cent of mercury, with an additional 2.27 per cent for the maximum, or a total of 67.34 per cent. Engelhardt and Maddrel found 67.62 per cent. The mother-liquor, evaporated at the ordinary temperature over sulphuric acid, gave further crystals containing 58.2 per cent of mercury, of which 26.2 was the minimum and 32.0 the maximum. The second mother-liquor gave more crystals, still richer in mercuric lactate (mercury at the minimum 48.3 per cent, at maximum 7.3 per cent).

These two last mixtures of lactates are soluble in water. We might perhaps succeed in preparing by fractional crystallisation a mercuric lactate entirely free from mercurous lactate; but the operation would not be very successful as the returns would be excessively small.

However, these experiments show that the lactates, prepared by boiling dilute lactic acid with oxide of mercury, are not definite compounds, but mixtures.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 15.

## THE MEASUREMENT OF TEMPERATURE BY ELECTRICAL MEANS.

ACCURACY of measurement in all scientific and commercial work, whether chemical, electrical, physical, or what not, is of the utmost importance.

Balances, micrometers, telescopes, &c., have all been brought to a very high pitch of perfection, but the measurement of temperatures, except within rather narrow limits, bounded on one side by the boiling-point of mercury, has been, until quite recently, very largely a matter of guesswork and conjecture.

In metallurgical and all furnace work, for instance, the question of temperature has depended upon the eye of the most experienced workman, and when this particular individual left, the whole process under his control ran the risk of being completely upset until another man acquired the necessary experience.

In many processes, such as annealing, it is important that the temperature should be known within two or three degrees. In an article on "Riddles Wrought in Iron and Steel," Mr. P. Kreuzpointer (*Cassier's Magazine*, 1901, pp. 276—280) says:—"We are comprehending that iron and steel are not the rigid immovable mass that we were wont to consider these metals; that they are, in fact, like very sensitive creatures, liable to be influenced by changes of temperature, unwilling to be abused and ill-treated. It is the degree of this changeability of steel which to-day requires our closest attention and study; the condition under which it changes, the reason why it changes, and what changes make a given grade of steel suitable or unsuitable for a given purpose. . . . We must use the pyrometer more freely than we do now in order to be able to determine the proper temperature at which to anneal and to temper, and to produce the same results uniformly at all times."

For many years the Cambridge Scientific Instrument Company have been experimenting with the electrical thermometers devised by Professor H. L. Callendar and Dr. E. H. Griffiths, in order to render them capable of standing the rough treatment of commercial work, and without sacrificing their well-known scientific accuracy, and they have now produced thermometers which fulfil these requirements.

The electrical thermometers made by this firm are of two kinds:—

I. *Resistance thermometers*, depending on the way in

which the resistance of a coil of wire is affected by its temperature.

II. *Thermo-electrical thermometers*, depending for their indications on the current generated by heating one of a pair of metallic junctions.

In the resistance thermometers, a fine platinum wire is wound on a mica frame; this wire is connected by means of stouter wires to the indicator or recorder. The platinum coil is protected from the action of fumes and from mechanical damage by means of a brass, steel, nickel, or porcelain tube, depending on the temperature it is required to measure. The thermometers can be placed in positions where it would be absolutely impossible to use or read mercury ones, and by means of compensating leads the indicators, where the temperature is read, can be placed at a considerable distance away, and by means of a switch-board any number of furnaces can be connected with the indicator, situated, say, in the manager's office. Again, by means of a Callendar recorder, a continuous record of the temperature can be obtained on a sheet of ruled paper.

Thermo-electrical thermometers are largely employed on the Continent for smelting, distilling, foundry-work, &c.; they will measure any temperature up to 1600° C. The couple consists of a platinum, platinum-iridium junction enclosed in a porcelain tube, and the temperature obtained is read on a special form of voltmeter, each division on the dial being equal to 10° C.; these thermometers, though not so accurate as the resistance ones, are useful for employment in places where there is a great risk of the destruction of the thermometers, their cost being less than that of the resistance ones, but as they require more frequent standardising they are not so trustworthy as resistance thermometers.

Both the above forms can be used for very wide ranges, or can be made for special purposes; they are suitable alike for cold storage rooms, for vats, stills, furnaces, &c., and if care be taken, it may be stated as a general rule that the temperature measurements can be relied upon to 0.5 per cent.

Among the advantages possessed by these instruments we may mention briefly the following:—They can be used for the measurement of very high or very low temperatures, beyond the range of the mercury thermometer; they can be placed in positions where it is impossible to use other kinds; they are very sensitive and accurate; their indications can be read at a considerable distance without appreciable error. In conjunction with a Callendar recorder, they will give a complete record of the temperature for a day or a week; if the temperature is read off on a Whipple indicator, no corrections are necessary, as the readings are direct.

## THE PRODUCTION OF FERROCYANOGEN FROM GAS-LIQUORS.

By ED. DONATH.

THE method of separating cyanogen known as the wet-method, certainly enables the cyanogen present in the gas to be recovered in a more complete manner than by any other method used up to the present. At the same time, the drawbacks resulting from the presence of cyanogen in the gas-liquors are got rid of, cyanogen being very prejudicial to the elimination of the sulphuretted hydrogen.

However, the old method of purifying the gas is still used, a process by which the cyanogen compounds and the sulphuretted hydrogen are removed simultaneously, and which makes the spent purifying material still the principal source of ferrocyanogen compounds and consequently of cyanogen compounds,

It is true that the amount of cyanogen present in gas-liquors is very small; nevertheless their recovery has occupied the attention of practical men for a long time past. Thus Mr. Bower, in his patent taken out in 1882, recommended the transformation of the cyanides in gas-liquors into ferrocyanides, before distillation with lime, by the addition of iron, and the recovery of these latter salts. In his well-known treatise on "Coal Tar and Ammonia" (3rd edition, page 527), G. Lunge speaks of this patent in an unfavourable manner. However, Mr. Bower's idea has often come up again, and we find in the *Chemische Industrie*, 1897, page 507, the description of a patent by the same H. Bower (English Patent, No. 361, January 6th, 1896), a patent which also has for its object the formation of double cyanides by the addition of iron or of a salt of iron to gas-liquors. These facts have decided me to describe some experiments I undertook in a manufactory of ammonia and ammoniacal salts.

We can see easily that gas-liquors exercise a corrosive action on iron, but this action apparently is more energetic when the water is heated. While the pumps which are used to pump up the cold ammoniacal liquor are corroded but slightly, it is by no means uncommon to observe strong corrosive action on the iron portions of the distilling apparatus. I have even seen, in the works in question, a Feldmann distilling apparatus which had become so completely friable that at many places it was easy to pierce the fairly thick sides with an ordinary knife. A portion removed with a knife consisted principally of graphite and Prussian blue, and it is highly probable that this latter body was formed, thanks to the presence of sulphides in the gas liquors, by the same process as in the purifying material.

In the same works sal-ammoniac was produced for some time by the direct saturation of the ammoniacal waters by raw hydrochloric acid. To precipitate the arsenic and the iron contained in the hydrochloric acid used, the latter was previously treated with a certain quantity of the yellow mother-liquors from the crystallisation, at a low temperature, of the carbonate of ammonium contained in the ammoniacal liquor. These mother-liquors thus contained the most easily soluble ammoniacal compounds; that is to say, the sulphides, the cyanides, and, as was to be expected, the ferrocyanides. It is well understood that this operation was accompanied by a considerable evolution of heat, and sulphuretted hydrogen was given off, but at the same time a dark green mud was formed in quantities by no means negligible. I had occasion to examine this mud, the result of several operations. In the dry state it consisted of a greenish-black powder, with a tarry odour, and was formed principally of Prussian blue accompanied by sulphide of arsenic and tarry products.

After several fruitless attempts, I succeeded in finding a method for the separation and preparation in a state of purity of the Prussian blue contained in this mud. I digested the dry mud with a certain quantity of raw, concentrated hydrochloric acid, which only dissolved the Prussian blue, and left the sulphide of arsenic and the tarry products intact. The hydrochloric solution was filtered through sheep's wool, and it was sufficient to dilute the filtered liquor to completely precipitate the Prussian blue.

The facts of which I have just spoken would hardly be of any commercial value by themselves; but when sal ammoniac is prepared in the manner described, that is to say by the direct saturation of the concentrated ammoniacal liquors, we can recover the cyanogen compounds contained in these liquors, seeing that they gradually accumulate in the yellow mother-liquors referred to above. This recovery can be effected without much difficulty, as the raw, concentrated hydrochloric acid, used for the extraction of the Prussian blue from the mud, will still serve for the preparation of the sal-ammoniac.—*Journ. für Gasbeleuchtung und Wasserversorgung*, vol. xlvi., p. 880.

CONTRIBUTIONS TO OUR KNOWLEDGE OF  
YTTERBIUM.\*

By ASTRID CLEVE.

(Continued from p. 263).

III. COMPOUNDS OF YTTERBIUM (continued).

Ytterbium-gold Chloride,  $\text{YbCl}_3 \cdot \text{AuCl}_3 \cdot 9\text{H}_2\text{O}$ .

CRYSTALLISES in light yellow, transparent, six-sided, deliquescent tables, if a solution of the calculated quantities of ytterbium chloride and acid gold chloride are strongly concentrated over sulphuric acid.

Analysis of the Pressed Salt.

- 0.7633 grm. of the substance gave 0.5281 grm.  $\text{Yb}_2\text{SO}_4 + \text{Au}$ , of which 0.1198 grm. was Au.
- 0.4259 grm. gave up 0.0552 grm. of water at  $100^\circ$ . From the residue 0.2949 grm. of  $\text{Yb}_2\text{SO}_4 + \text{Au}$  was obtained.
- 0.2478 grm. of the substance gave 0.3500 grm.  $\text{AgCl} + \text{Au}$ , of which 0.0651 grm. was Au.

|                      | Calculated for<br>$\text{YbCl}_3 \cdot \text{AuCl}_3 \cdot 9\text{H}_2\text{O}$<br>in per cent. |       | Found. |   |       |
|----------------------|-------------------------------------------------------------------------------------------------|-------|--------|---|-------|
|                      | I.                                                                                              | II.   | III.   |   |       |
| Yb ..                | 173.1                                                                                           | 23.26 | 23.47  | — | —     |
| Au ..                | 197.2                                                                                           | 26.50 | 26.2   | — | 26.3  |
| Cl <sub>6</sub> ..   | 212.7                                                                                           | 28.58 | —      | — | 28.43 |
| 9H <sub>2</sub> O .. | 161.18                                                                                          | 21.66 | —      | — | —     |

|                                         |        |        |       |   |  |
|-----------------------------------------|--------|--------|-------|---|--|
|                                         | 744.18 | 100.00 |       |   |  |
| $\text{Yb}_2\text{SO}_4 + \text{Au}$ .. | 69.12  | 69.19  | 69.24 | — |  |

About 5 molecules of water are lost at  $100^\circ$ ; the calculated loss of weight is 12.11 per cent, and that found (II.) 12.91 per cent.

The erbium compound,  $\text{ErCl}_3 \cdot \text{AuCl}_3 \cdot 9\text{H}_2\text{O}$  (Cleve, *Bull. Soc. Chim.*, 1874, xxi., 345), has an exactly similar composition, while the gadolinium (Benedicks, *Zeit. Anorg. Chem.*, xxii., 404) and praseodymium (v. Schéele, "Om Praseodym, &c." Inaug. Diss., Upsala, 1900, p. 47) compounds contain 10 molecules of water, and the ytterbium-gold chloride (*Bull. Soc. Chim.*, 1872, xviii., 198) belongs to the different type  $\text{R}^{III}\text{Cl}_3 \cdot 2\text{AuCl}_3 \cdot 16\text{H}_2\text{O}$ .

Specific Gravity and Molecular Volume.

|                                     |       |
|-------------------------------------|-------|
| 1. 0.9608 grm., specific gravity .. | 2.857 |
| 2. 0.9296 " " " ..                  | 2.843 |
| Mean value ..                       | 2.850 |
| Molecular volume ..                 | 261.  |

Ytterbium-platinum Cyanide,  $2\text{Yb}(\text{CN})_3 \cdot 3\text{Pt}(\text{CN})_2 \cdot 18\text{H}_2\text{O}$ .

This salt was prepared from ytterbium sulphate and barium-platinum cyanide. It crystallises similarly to the corresponding yttrium compound in perfectly formed prisms, which appear a brilliant red by transmitted light, and green and bluish violet respectively by reflected light. The water of crystallisation was found to amount to 18 molecules, and not to 21, as in the case of the yttrium and erbium salts, according to Cleve (*Bull. Soc. Chim.*, 1872, xviii., 198). Thus the ytterbium compound agrees exactly with that of gadolinium (Benedicks, *loc. cit.*, 406). When dried over sulphuric acid, 16 molecules of water are slowly given up. At  $100^\circ$  the efflorescence of the crystals proceeds more quickly, but 2 molecules of  $\text{H}_2\text{O}$  are still held in combination. When 14H<sub>2</sub>O have passed off the red colour disappears, and the powder becomes brick-yellow and then whitish. It is easily soluble, and resists the action of the air.

Analysis of the Pressed Salt.

- 0.7110 grm. of the substance dried over sulphuric acid suffered a loss of weight of 0.1358 grm.
- 0.3457 grm. of the substance gave 0.2676 grm.  $\text{Yb}_2\text{SO}_4 + \text{Pt}$ , of which 0.1273 grm. was Pt.

\* From the *Zeitschrift für Anorganische Chemie*, xxxii.

|                                         | Calculated for<br>$2\text{Yb}(\text{CN})_3 \cdot 3\text{Pt}(\text{CN})_2 \cdot 18\text{H}_2\text{O}$<br>in per cent. |        | Found. |       |
|-----------------------------------------|----------------------------------------------------------------------------------------------------------------------|--------|--------|-------|
|                                         | I.                                                                                                                   | II.    | I.     | II.   |
| Yb <sub>2</sub> ..                      | 346.2                                                                                                                | 22.09  | —      | 22.15 |
| Pt <sub>3</sub> ..                      | 584.4                                                                                                                | 37.28  | —      | 36.83 |
| (CN) <sub>12</sub> ..                   | 312.48                                                                                                               | 19.93  | —      | —     |
| 16H <sub>2</sub> O ..                   | 288.32                                                                                                               | 18.40  | 19.06  | —     |
| 2H <sub>2</sub> O ..                    | 36.04                                                                                                                | 2.30   | —      | —     |
|                                         | 1567.44                                                                                                              | 100.00 |        |       |
| $\text{Yb}_2\text{SO}_4 + \text{Pt}$ .. | 77.76                                                                                                                |        |        | 77.41 |

Specific Gravity and Molecular Volume.

- 1.0036 grms. (large crystals), sp. gr. 2.661
- 1.1911 " (small crystals), " 2.657
- 1.2100 " (small crystals), " 2.658

|                     |       |
|---------------------|-------|
| Mean value ..       | 2.659 |
| Molecular volume .. | 589.5 |

Potassium-ytterbium Ferrocyanide,  $\text{KYb}(\text{CN})_6 \cdot \text{Fe} \cdot 3\text{H}_2\text{O}$   
(Dried over Sulphuric Acid).

Potassium ferrocyanide gives with solutions of ytterbium chloride the above double salt as a white, fine, granular precipitate, somewhat soluble in excess of the ferrocyanide. After having been dried it has a blue-green tinge, and in the desiccator it retains three molecules of water, one of which it loses at  $100^\circ$ .

Analysis.

- 0.6172 grm. of the substance dried over sulphuric acid suffered a loss in weight of 0.0218 grm. at  $100^\circ$ .
- 0.4546 grm. of the substance dried over sulphuric acid gave, after ignition and extraction with water, 0.2632 grm.  $\text{Yb}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ .

|                                                    | Calculated for<br>$\text{KYb}(\text{CN})_6 \cdot \text{Fe} \cdot 3\text{H}_2\text{O}$<br>in per cent. |      | Found. |     |
|----------------------------------------------------|-------------------------------------------------------------------------------------------------------|------|--------|-----|
|                                                    | I.                                                                                                    | II.  | I.     | II. |
| $\text{Yb}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ .. | 57.90                                                                                                 | —    | 57.98  | —   |
| 1 mol. H <sub>2</sub> O ..                         | 3.76                                                                                                  | 3.53 | —      | —   |

- 0.2331 grm. of the substance dried at  $110^\circ$  gave 0.1409 grm.  $\text{Yb}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ , from which 0.0994 grm.  $\text{Yb}_2\text{O}_3$  was precipitated from the almost neutral solution by means of oxalic acid. 0.0359 grm. of KCl was obtained from the filtrate.

|                      | Calculated for<br>$\text{KYb}(\text{CN})_6 \cdot \text{Fe} \cdot 2\text{H}_2\text{O}$<br>in per cent. |        | Found. |     |
|----------------------|-------------------------------------------------------------------------------------------------------|--------|--------|-----|
|                      | I.                                                                                                    | II.    | I.     | II. |
| Yb ..                | 173.1                                                                                                 | 37.58  | 37.45  | —   |
| K ..                 | 39.15                                                                                                 | 8.50   | 8.08   | —   |
| Fe ..                | 56.10                                                                                                 | 12.16  | 12.46  | —   |
| (CN) <sub>6</sub> .. | 156.24                                                                                                | 33.92  | —      | —   |
| 2H <sub>2</sub> O .. | 36.04                                                                                                 | 11.74  | —      | —   |
|                      | 460.53                                                                                                | 100.00 |        |     |

Corresponding double ferrocyanides of yttrium, erbium, and samarium are known (Cleve).

Ytterbium Nitrates.

1.  $\text{Yb}_3\text{NO}_8 \cdot 3\text{H}_2\text{O}$ .

If a solution of ytterbium nitrate is kept for some months over sulphuric acid, large transparent perfectly-formed tables of the above compound separate out. They are deliquescent.

Analysis of the Pressed Salt.

- 0.6418 grm. of the substance gave, after cautious heating and ignition, 0.3057 grm.  $\text{Yb}_2\text{O}_3$ .

|                      | Calculated for<br>$\text{Yb}_3\text{NO}_8 \cdot 3\text{H}_2\text{O}$ in per cent. |        | Found. |     |
|----------------------|-----------------------------------------------------------------------------------|--------|--------|-----|
|                      | I.                                                                                | II.    | I.     | II. |
| Yb ..                | 173.1                                                                             | 41.88  | 41.83  | —   |
| 3NO <sub>3</sub> ..  | 186.12                                                                            | 45.04  | —      | —   |
| 3H <sub>2</sub> O .. | 54.06                                                                             | 13.08  | —      | —   |
|                      | 413.28                                                                            | 100.00 |        |     |



2.  $\text{Yb}_3\text{NO}_3 + 4\text{H}_2\text{O}$ .

Crystallises from concentrated nitric acid in transparent deliquescent prisms. The salt may be obtained, though with more difficulty, by strongly concentrating the aqueous solution; it then forms fine needles.

## Analysis of the Pressed Salt.

(a). Crystallised from concentrated nitric acid.

1. 1.0652 grm. of the substance gave 0.4845 grm.  $\text{Yb}_2\text{O}_3$ .
2. 0.5225 grm. of the substance gave 0.2392 grm.  $\text{Yb}_2\text{O}_3$ .
3. 0.5141 grm. of the substance gave 80.04 c.c. NO at 0° and 760 m.m. mercury pressure by Schulze's method (*Zeit. Anal. Chem.*, ix., 400).

(b). Crystallised from water.

4. 0.5547 grm. of the substance gave 0.2505 grm.  $\text{Yb}_2\text{O}_3$ .

|                      | Calculated for<br>$\text{Yb}_3\text{NO}_3 + 4\text{H}_2\text{O}$ ,<br>in per cent. |       | Found. |       |       |       |
|----------------------|------------------------------------------------------------------------------------|-------|--------|-------|-------|-------|
|                      |                                                                                    |       | I.     | II.   | III.  | IV.   |
| Yb ..                | 173.1                                                                              | 40.14 | 39.95  | 40.19 | —     | 39.66 |
| 3NO ..               | 90.12                                                                              | 20.89 | —      | —     | 20.89 | —     |
| O <sub>8</sub> ..    | 96                                                                                 | 22.26 | —      | —     | —     | —     |
| 4H <sub>2</sub> O .. | 72.08                                                                              | 16.71 | —      | —     | —     | —     |

431.3 100.00

**Specific Gravity and Molecular Volume.**—0.7066 grm. of the salt crystallised from nitric acid. Specific gravity, 2.682; molecular volume, 160.9.

All other known nitrates of rare earths contain more water of crystallisation than these two ytterbium compounds, e.g., the yttrium and gadolinium salts crystallise with 5H<sub>2</sub>O (Benedicks, *loc. cit.*, p. 407), and the samarium (Cleve, *loc. cit.*, p. 15) and praseodymium (v. Schöele, *loc. cit.*, p. 52) salts with 6H<sub>2</sub>O.

Ytterbium Sulphate,  $\text{Yb}_2.3\text{SO}_4 + 8\text{H}_2\text{O}$ .

The sulphate has been prepared in the pure state by Nilson and analysed (*loc. cit.*, p. 10). In the cold the salt is considerably more soluble than at higher temperatures, and hence is precipitated on heating cold concentrated solutions.

Crystals prepared in this way contain the same amount of water as the salt which separates out on spontaneous evaporation. The reaction is neutral in solutions which are not too dilute. When very dilute, as will be shown later, hydrolysis begins.

## Analysis of the Pressed Substance.

- (a). Crystallised at the temperature of the room. 2.2041 grms. of the salt gave 1.9779 grms.  $\text{BaSO}_4$ .
- (b). Crystallised at the temperature of ebullition. 1.4054 grms. of the substance gave up 0.2617 grm. H<sub>2</sub>O when dried at about 200°.

|                            | Calculated for<br>$\text{Yb}_2.3\text{SO}_4 + 8\text{H}_2\text{O}$ ,<br>in per cent. |        | Found. |       |
|----------------------------|--------------------------------------------------------------------------------------|--------|--------|-------|
|                            |                                                                                      |        | a.     | b.    |
| $\text{Yb}_2\text{O}_3$ .. | 394.2                                                                                | 50.63  | —      | —     |
| $3\text{SO}_3$ ..          | 240.18                                                                               | 30.85  | 30.77  | —     |
| $8\text{H}_2\text{O}$ ..   | 144.16                                                                               | 18.52  | —      | 18.62 |
|                            | 778.54                                                                               | 100.00 |        |       |

## Specific Gravity and Molecular Volume.

1. 1.2997 grms., temperature 20.6°, sp. gr. 3.30
2. 1.6337 grms., temperature 18.3°, sp. gr. 3.26

Mean value .. .. 3.28  
Molecular volume .. .. 237

Anhydrous Ytterbium Sulphate,  $\text{Yb}_2.3\text{SO}_4$ .

1. 1.2292 grms., specific gravity .. .. 3.627
2. 1.0947 " " " " " " 3.617

Mean value .. .. 3.62  
Molecular volume .. .. 175

Determinations of the solubility of the sulphate were made as follows:—The finely powdered anhydrous salt was gradually introduced into a vessel of water until it was present in excess, and was kept in suspension for two to three hours by means of a stirrer. It was kept at the required temperature on a water-bath. Then 25 c.c. of the solution were drawn off, evaporated in a platinum dish, and the residue weighed as anhydrous sulphate.

|    | Temp. | C.c. solution.       | Grms. weight | Grms. $\text{Yb}_2.3\text{SO}_4$ gave |
|----|-------|----------------------|--------------|---------------------------------------|
| 1. | 0°    | (a) 25,              | 35.341,      | 10.8246                               |
|    |       | (b) 25,              | 35.340,      | 10.8317                               |
| 2. | 15.5° | 25,                  | 32.490,      | 8.3521                                |
| 3. | 35°   | 25,                  | 29.490,      | 4.7393                                |
| 4. | 55°   | 25,                  | 27.572,      | 2.8540                                |
| 5. | 60°   | 25,                  | 27.215,      | 2.5556                                |
| 6. | 70°   | 25,                  | 26.410,      | 1.7796                                |
| 7. | 80°   | 25,                  | 26.265,      | 1.7038                                |
| 8. | 90°   | (a) 25,              | 25.914,      | 1.3968                                |
|    |       | (b) 25,              | 25.930,      | 1.4593                                |
| 9. | 100°  | 27.676 grm. solution |              | 1.2346                                |

According to these experiments, 100 parts of water dissolve—

|         |                                                                 |
|---------|-----------------------------------------------------------------|
| At 0°,  | 44.2 parts $\text{Yb}_2.3\text{SO}_4$ (mean of 44.15 and 44.21) |
| " 15.5° | 34.6 " "                                                        |
| " 35°   | 19.1 " "                                                        |
| " 55°   | 11.5 " "                                                        |
| " 60°   | 10.4 " "                                                        |
| " 70°   | 7.22 " "                                                        |
| " 80°   | 6.93 " "                                                        |
| " 90°   | 5.83 " (mean of 5.70 and 5.96)                                  |
| " 100°  | 4.67 " "                                                        |

These data show that ytterbium sulphate in the cold possesses a fairly high solubility, which decreases with rising temperature—rapidly to about 70°, and then more slowly. The salt is always considerably more soluble than the sulphates of yttrium and gadolinium; of the compounds most closely allied to it, erbium sulphate surpasses it most in this respect. However, the solubility of the latter is not accurately known, as it was found from material containing ytterbium (Cleve and Höglund, *Bih. K. Vet. Ak. Handl. Stockholm*, 1873, i., No. 8). Some data relating to this point may be given for comparison.

- 100 parts H<sub>2</sub>O dissolve at 0° 3.98 parts  $\text{Gd}_2.3\text{SO}_4$  (Benedicks, *loc. cit.*, p. 409).
- 100 parts H<sub>2</sub>O dissolve at temperature of room 43 parts  $\text{Er}_2.3\text{SO}_4$ .
- 100 parts H<sub>2</sub>O dissolve at temperature of room 1.52 parts  $\text{Y}_2.3\text{SO}_4$ .
- 100 parts H<sub>2</sub>O dissolve at 100° 20 parts  $\text{Er}_2.3\text{SO}_4 + 8\text{H}_2\text{O}$ .
- 100 parts H<sub>2</sub>O dissolve at 100° 4.8 parts  $\text{Y}_2.3\text{SO}_4 + 8\text{H}_2\text{O}$ .

Ytterbium Sulphite,  $\text{Yb}_2.3\text{SO}_3 + 9\text{H}_2\text{O}$ .

It was prepared by the decomposition, by means of  $\text{SO}_2$ , of freshly precipitated carbonate suspended in water. After standing some time over sulphuric acid, the solution, which is filtered if necessary, deposits a white felt-like crust, insoluble in water.

## Analysis of the Freshly-prepared Pressed Salt.

- 0.3490 grm. of the substance gave, after oxidation with chlorine water and precipitation with ammonia, 0.1840 grm.  $\text{Yb}_2\text{O}_3$ . 0.3189 grm.  $\text{BaSO}_4$  was obtained from the filtrate.

|                            | Calculated for<br>$\text{Yb}_2.3\text{SO}_3 + 9\text{H}_2\text{O}$ , in per cent. |        | Found. |
|----------------------------|-----------------------------------------------------------------------------------|--------|--------|
| $\text{Yb}_2\text{O}_3$ .. | 394.2                                                                             | 52.56  | 52.72  |
| $3\text{SO}_2$ ..          | 192.18                                                                            | 25.67  | 25.07  |
| $9\text{H}_2\text{O}$ ..   | 162.18                                                                            | 21.67  | —      |
|                            | 748.56                                                                            | 100.00 |        |

Yttrium, erbium, and samarium sulphites all crystallise with 3H<sub>2</sub>O, and the didymium salt with 6H<sub>2</sub>O (Cleve).

*Ytterbium Hyposulphate.*

A solution of the salt, prepared by double decomposition of ytterbium sulphate and barium hyposulphate, thickens on concentration over sulphuric acid to a syrupy consistency, and crystals form in rays. On keeping it in closed tubes, it decomposes after some time, giving up  $\text{SO}_2$ .

*Ytterbium Ethyl Sulphate,  $\text{Yb}_2\text{C}_2\text{H}_5\text{SO}_4 \cdot 9\text{H}_2\text{O}$ .*

Large, transparent, readily-soluble crystals, obtained from ytterbium sulphate and barium ethyl sulphate. It resists the action of the air; the neutral solution also is stable, but decomposition soon begins in feebly acid solutions. At  $70^\circ$ , alcohol begins to escape; at  $160^\circ$ , it is completely given off at a more rapid rate. Six molecules of water are given up over sulphuric acid.

*Analysis of the Pressed Salt.*

1. 1.1627 grms. of the substance slowly lost in the desiccator 0.1775 grm., and suffered a further loss of 0.2310 grm. at  $160^\circ$ , and gave 0.5216 grm.  $\text{Yb}_2\text{C}_2\text{H}_5\text{SO}_4$ .
2. 0.7928 grm. of the substance gave up 0.1201 grm. of water in the desiccator, and yielded 0.3552 grm.  $\text{Yb}_2\text{C}_2\text{H}_5\text{SO}_4$ .

|                                    | Calculated for<br>$\text{Yb}_2\text{C}_2\text{H}_5\text{SO}_4 \cdot 9\text{H}_2\text{O}$ ,<br>in per cent. |        | Found.                            |       |
|------------------------------------|------------------------------------------------------------------------------------------------------------|--------|-----------------------------------|-------|
|                                    |                                                                                                            |        | I.                                | II.   |
| Yb .. ..                           | 173.1                                                                                                      | 24.36  | 24.41                             | 24.45 |
| $\text{C}_2\text{H}_5\text{OH}$ .. | 138.18                                                                                                     | 19.45  | 19.86                             | —     |
|                                    |                                                                                                            |        | (Loss of wt.<br>at $160^\circ$ ). |       |
| $3\text{H}_2\text{SO}_4$ ..        | 291.21                                                                                                     | 40.98  | —                                 | —     |
| $6\text{H}_2\text{O}$ ..           | 108.12                                                                                                     | 15.21  | 15.27                             | 15.15 |
|                                    |                                                                                                            |        | (Loss of wt.<br>in desiccator).   |       |
|                                    | 710.61                                                                                                     | 100.00 |                                   |       |

*Specific Gravity and Molecular Volume.*—1.1477 grms. Specific gravity, 2.024; molecular volume, 351.  
(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

*Ordinary Meeting, November 6, 1902.*

Professor McLROD, F.R.S., Vice-President, in the Chair.

(Concluded from p. 265).

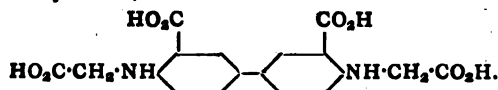
152. "Di-indigotin." By J. MOIR, M.A., D.Sc.

The author's object in the following experiments was to prepare the diphenyl analogue of indigotin, in order to ascertain what influence the doubling of the molecule would have on the colour, since this exercises a marked influence in the case of the benzidine dyes as compared with ordinary azo-dyes derived from aniline. Although successful in preparing the new substance, the yield was so poor and its properties so troublesome to investigate that only a meagre description of it is possible.

The method consisted in applying the synthesis of indigo employed by F. Baeyer and Co. to benzidine-dicarboxylic acid instead of to anthranilic acid.

4:4'-Benzidine-3:3'-dicarboxylic acid was prepared from *o*-nitrobenzoic acid by reduction with sodium hydroxide and aluminium powder until the solution, which was at first dark red, became paler by the conversion of azo- to hydrazo-acid; it was then precipitated with excess of acetic acid, and the hydrazo-acid thus obtained boiled with strong hydrochloric acid to convert it into the benzidine derivative, which was obtained on dilution as an almost insoluble powder, coloured pale green

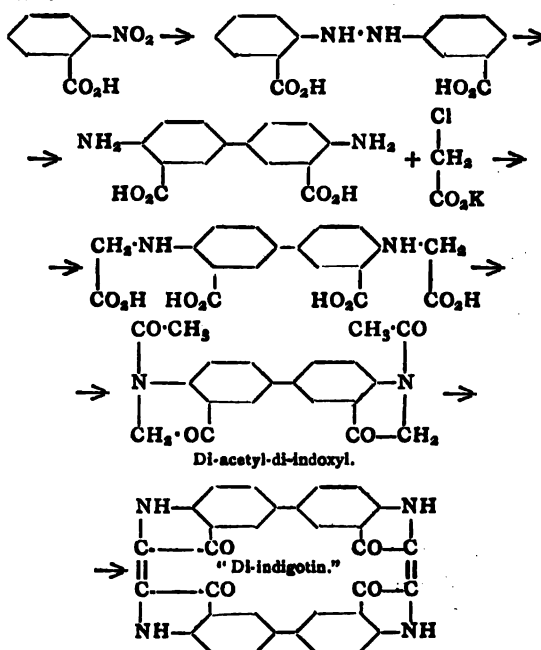
by some oxidation product which could not be removed. It was dissolved in the smallest possible quantity of potassium carbonate solution and mixed with excess of neutral potassium monochloracetate solution. On boiling the mixture for some hours, a small quantity of a crystalline precipitate was obtained, which was filtered off and dried. It melted above  $300^\circ$  and is probably bisphenylglycine-*o*-carboxylic acid,—



This was boiled during four hours with excess of acetic anhydride and dry potassium acetate, and finally taken to dryness on the water-bath. This process closes the indoxyl ring. The product freed from inorganic substances by washing, on digestion with dilute sodium hydroxide, dissolved, the solution becoming green, whilst a pellicle of the di-indigotin formed on the surface. Air was passed into the diluted solution, and the olive-green substance was filtered off, washed, and dried. The filtrate was also dark green and gave on acidifying a dark green carboxylic acid, which probably results from the formation of the indoxyl ring at one end only of the first substance.

The properties of the di-indigotin precluded any exact investigation. It is only very slightly soluble in boiling  $\beta$ -toluidine, giving a solution distinctly bluer than the original substance as first prepared in fine suspension. It gives an olive-green solution in concentrated sulphuric acid, but is apparently not sulphonated at  $100^\circ$ . It was not analysed, because it was impossible to guarantee its uniformity, but its stability precludes the possibility that it is merely a substituted indigotin.

The following scheme probably represents its formation:—



An attempt to confirm this by obtaining the same substance by another synthesis was made. All the methods involving potash fusion failed, but the method of Blank (*Ber.*, 1898, xxxi., 2816) was partially successful, and is here given as involving the preparation of a new substance. Benzidine was heated with a solution of half its weight of diethyl bromomalonate in chloroform until the

odour of the former had gone; the product was boiled out with chloroform and benzene, and the filtered extracts concentrated and cautiously treated with ligroin, when the new ester crystallised out in plates. After two recrystallisations from ethyl acetate, it was white and melted sharply at 138°. An analysis showed it to be the expected *ethyl benzidino dimalonate*.—



o'1564 gave o'3542 CO<sub>2</sub> and o'0918 H<sub>2</sub>O. C=61'73; H=6'52.

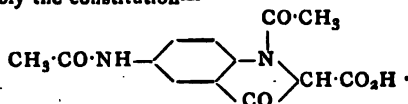
o'2562 gave 12'80 c.c. moist nitrogen at 21° and 759 m.m. N=5'69.

C<sub>26</sub>H<sub>32</sub>O<sub>8</sub>N<sub>2</sub> requires C=62'40, H=6'40, N=5'60 %.

It forms minute lustrous plates, sparingly soluble in alcohol and in cold benzene, easily so in hot benzene, ethyl acetate, and chloroform. It is slowly hydrolysed by alkalis, the solution giving, with acids, a flocculent precipitate of the corresponding acid.

On the analogy of Blank's indigo synthesis from ethyl anilinomalonate, the new ester was heated to 200° in an oil-bath until the evolution of vapour ceased. The product was dissolved in a little acetone, filtered, and precipitated by alcohol, giving a dark gum (whilst the alcoholic solution gave a little of the unchanged substance in needles, identified by mixed melting-point). On boiling the gum with alcohol, a red resin was left, and the alcoholic solution was concentrated and hydrolysed by sodium hydroxide, giving a deep green solution, but no other evidence of the presence of di-indigotin in solid form. Evidently the action of heat on ethyl benzidino-dimalonate is not analogous to its action on ethyl anilinomalonate, for many other temperatures than 200° were tried, but with even less success.

The author has prepared another new substance by boiling benzidine dicarboxylic acid with dry chloroacetic acid and dry potassium chloracetate; carbon dioxide was split off, but the product was acetylated as in the former synthesis. A crystalline carboxylic acid (lance-shaped white crystals, m.p. 292°) was isolated and re-crystallised from boiling ethyl acetate. Analysis showed C=64'94, H=4'85, N=7'91, hence the formula C<sub>19</sub>H<sub>16</sub>O<sub>5</sub>N<sub>2</sub>, and probably the constitution—



153. "Note on the Localisation of Phosphates in the Sugar-cane." By C. H. G. SPRANKLING.

As very little work has been done with regard to the position of mineral constituents in plants, the author selected the sugar-cane as a convenient plant to ascertain the position of the phosphates therein.

Equal portions of three sound canes were thoroughly dried, and after being ground to a fine powder, weighed quantities were ignited, using a slight modification of Fluckiger's method (*Zeit. Anal. Chem.*, 1889, xxvii., 637).

The ash in each case was extracted with dilute nitric acid, and the final ignited residue taken to be silica; the phosphates in the nitric acid solution were estimated.

From the results of his experiments, the author draws the following conclusions:—

(a). Phosphates are immediately absorbed by the roots of the plant.

(b). These phosphates are rapidly transferred to the upper parts of the plant.

(c). A quantity of phosphates is stored in the upper portions and leaves of the cane.

(d). The silica is transferred regularly to the leaves, and there stored.

154. "On the Non-existence of the Gaseous Sulphide of Carbon described by Deninger." By E. J. RUSSELL and N. SMITH.

The authors have examined the gas prepared as Deninger describes (*Journal für Prakt. Chemie*, 1895, [2], li., 346), and to which he gave the formula CS. Deninger prepared it (1) by heating together in a sealed tube chloroform and sodium sulphide, (2) by subjecting to the same treatment a mixture of iodoform and silver sulphide, (3) by the action of sodium on carbon disulphide mixed with aniline. The authors can find no evidence whatever of the existence of this gas. Sodium and carbon disulphide left in a closed apparatus fitted with a mercury gauge were found to produce no gaseous substance; a mixture of sodium, aniline, and carbon disulphide evolved hydrogen and hydrogen sulphide, and these gases in escaping carried over some carbon disulphide. Evidence that no other gas was present was obtained (1) by analysis, (2) by cooling the mixture and separating it into its constituents.

On heating together in a sealed tube to 180° a mixture of potassium sulphide and chloroform, the products were hydrogen sulphide, hydrogen chloride, chloroform, a reddish-yellow liquid which was apparently an alkyl sulphide, and free sulphur. A mixture of iodoform and silver sulphide similarly treated yielded hydrogen, carbon disulphide, and the reddish-yellow liquid. Although owing to the complex nature of these products the authors can hardly assert that no new gas having the formula CS was present, they could find no indication of any such compound.

155. "Hydroxyoxamides." Part II. By R. H. PICKARD, C. ALLEN, W. A. BOWDLER, and W. CARTER.

The authors described the following new hydroxyoxamides, namely:—*o*-, *m*-, and *p*-nitrophenyl-, *o*-tolyl-, and ethyl-hydroxyoxamides. These react as hydroxamic acids (*Trans.*, 1901, lxxix., 841). The esters of hydroxyoxamides are weak acids, and the hydroxyoxamides react with phenylhydrazine, forming phenylhydrazides. These and other reactions show that the hydroxyoxamides behave like typical hydroxamic acids, and are therefore constituted according to the formula R·NH·CO·C(OH):NOH and not R·NH·C:(NOH)·CO<sub>2</sub>H, as suggested by Schiff (*Annalen*, 1902, cccxxi., 357) and Hollemann (*Rec. Trav. Chim.*, 1896, xv., 148).

156. "Isometric Anhydrous Sulphates of the Form M"SO<sub>4</sub>.R<sub>2</sub>SO<sub>4</sub>." By F. R. MALLETT.

The author described the anhydrous salts MgSO<sub>4</sub>.K<sub>2</sub>SO<sub>4</sub>, MgSO<sub>4</sub>.Rb<sub>2</sub>SO<sub>4</sub>, MnSO<sub>4</sub>.K<sub>2</sub>SO<sub>4</sub>, MnSO<sub>4</sub>.Rb<sub>2</sub>SO<sub>4</sub>, MnSO<sub>4</sub>.Ti<sub>2</sub>SO<sub>4</sub>, NiSO<sub>4</sub>.K<sub>2</sub>SO<sub>4</sub>, and CoSO<sub>4</sub>.K<sub>2</sub>SO<sub>4</sub>, which all crystallise in tetrahedral forms, being allied in this respect to the sulphates 2M"SO<sub>4</sub>.R<sub>2</sub>SO<sub>4</sub> previously described (*Trans.*, 1900, lxxvii., 216). They vary in their stability when exposed to the air, some remaining unaltered, whilst others are converted somewhat rapidly into the hexahydrated salts, M"SO<sub>4</sub>.R<sub>2</sub>SO<sub>4</sub>.6H<sub>2</sub>O.

157. "The Catalytic Racemisation of Amygdalin." By J. W. WALKER.

Much more amygdalin is dissolved by water containing a small quantity of alkali, of alkaline earth, or of alkali carbonate, than by pure water. From polarimetric determinations of the rate of hydrolysis of the various substances involved, the author concludes that in alkaline solution the glucoside is racemised by the catalytic action of the hydroxyl ions, and that amygdalinic acid is racemoid with respect to its asymmetric carbon atom.

158. "On Asymmetric Optically Active Selenium Compounds and on the Sexavalency of Selenium and Sulphur." By W. J. POPE, F.R.S., and A. NEVILLE, B.Sc.

*Methylphenyl selenide*, C<sub>6</sub>H<sub>5</sub>·Se·CH<sub>3</sub>, the first mixed alkyl selenide which has been described, is obtained as a pale yellow oil boiling at 200–201° by the action of methyl iodide on the sodiophenyl selenide of Krafft and Lyons. Owing, apparently, to the highly basic character of selenium, it differs markedly from methylphenyl sulphide in that it combines readily with bromoacetic acid to form the asymmetric *methylphenylselenetins bromide*.—



this salt crystallises in colourless scales melting at  $111^{\circ}$ , and on treatment with silver *d*-bromocamphorsulphonate, yields a mixture of the two following salts, which may be separated by crystallisation from alcohol.

*d*-Methylphenylselenetene *d*-bromocamphorsulphonate (*d*-B, *d*-A),  $\text{Se}(\text{CH}_3)(\text{C}_6\text{H}_5)(\text{CH}_2\cdot\text{CO}_2\text{H})\cdot\text{C}_{10}\text{H}_{14}\text{BrOSO}_3$ , is the less readily soluble, and forms needles melting at  $168^{\circ}$ ; in aqueous solution, its rotations are  $[\alpha]_D = +61\cdot26^{\circ}$  and  $[\text{M}]_D = +330\cdot8^{\circ}$ .

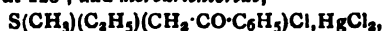
*l*-Methylphenylselenetene *d*-bromocamphorsulphonate (*l*-B, *d*-A), is much more soluble than the preceding salt, and is obtained in minute white scales melting at  $151^{\circ}$ . Its rotations in aqueous solution are  $[\alpha]_D = +38\cdot81^{\circ}$  and  $[\text{M}]_D = +209\cdot6^{\circ}$ . Since the *d*-bromocamphorsulphonic ion has the molecular rotation  $[\text{M}]_D = +270^{\circ}$ , the corresponding value for the optically active selenetene ion is  $[\text{M}]_D = \pm 60\cdot6^{\circ}$ .

The *dextro*- and *laevo*-methylphenylselenetene platini-chlorides,  $[\text{Se}(\text{CH}_3)(\text{C}_6\text{H}_5)(\text{CH}_2\cdot\text{CO}_2\text{H})\text{Cl}]_2\text{PtCl}_4$ , form yellow prisms melting at  $171^{\circ}$ ; they have the molecular rotations  $[\text{M}]_D = +55\cdot0^{\circ}$  and  $-54\cdot3^{\circ}$  respectively in acetone solution.

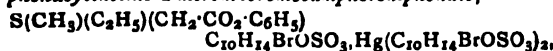
The same optically inactive methylphenylselenetene mercurioides,  $\text{Se}(\text{CH}_3)(\text{C}_6\text{H}_5)(\text{CH}_2\cdot\text{CO}_2\text{H})\cdot\text{HgI}_2$ , is obtained by precipitating either of the salts (*d*-B, *d*-A) (*l*-B, *d*-A), or methylphenylselenetene bromide with a solution of potassium mercuric iodide; the mercurioides forms white scales melting at  $141$ – $142^{\circ}$ . The optical inactivity of this salt being remarkable, in view of the fact that the *d*- and *l*-benzylphenylallylmethylammonium ions preserve their optical activity during formation of the mercurioides, it seemed desirable to ascertain if the same behaviour was exhibited during the formation of a mercurioides from an optically active thietine.

The authors therefore prepared the optically active methylethylphenacylthietine salts of Smiles instead of the methylethylthietine compounds previously described by Pope and Peachey, owing to the greater optical rotations exhibited by the optically active ions of the former substances. It was then found that the *d*-methylethylphenacylthietine *d*-bromocamphorsulphonate, (*d*-B, *d*-A), had the molecular rotation  $[\text{M}]_D = +332\cdot8^{\circ}$ , whilst the corresponding salt (*l*-B, *d*-A), gave  $[\text{M}]_D = +210\cdot6^{\circ}$ ; the optically active methylethylphenacylthietine ion therefore has the molecular rotation  $[\text{M}]_D = \pm 61\cdot1^{\circ}$ , a value about three times as great as  $[\text{M}]_D = \pm 19\cdot4^{\circ}$ , the molecular rotation deduced from Smiles' determinations.

Each of the above salts yields the same optically inactive mercurioides,  $\text{S}(\text{CH}_3)(\text{C}_2\text{H}_5)(\text{CH}_2\cdot\text{CO}\cdot\text{C}_6\text{H}_5)\cdot\text{HgI}_2$ , melting at  $128^{\circ}$ , and mercurichloride,—



melting at  $119^{\circ}$ . They also give the same methylethylphenacylthietine-*d*-mercuribromocamphorsulphonate,—



which has a molecular rotation  $[\text{M}]_D = +799^{\circ}$ , very nearly three times that of the *d*-bromocamphorsulphonic ion, and in which the sulphur atom does not seem to act as a centre of optical activity.

The authors conclude that the mercuri-compounds of the sulphonium and selenonium bases probably contain sesquivalent sulphur and selenium.

159. "The Transformation of Acetylchloroaminobenzenes into the Isomeric Chloroacetanilides." By F. D. CHATTAWAY and K. J. P. ORTON.

Owing to the recent publication by Blankema (*Proc. K. Akad. Wetensch. Amsterdam*, 1902, p. 178) of measurements of the velocity of transformation of acetylchloroaminobenzene into *p*-chloroacetanilide, the authors wish to record the results of some preliminary experiments made by them two years ago (September, 1900) on isomeric changes of this type. These experiments were not published at that time, as Armstrong had previously stated (*Trans.*, 1900, lxxvii., 1053) that he was engaged on a similar investigation.

To follow the course of the isomeric change, a 1 per cent solution of the chloramine in glacial acetic acid or in 50 per cent acetic acid was kept at a constant temperature in the dark, and the amount transformed estimated at intervals by titrating the iodine liberated from hydriodic acid by the unchanged substance. In the presence of an acid, such as hydrochloric or sulphuric, the isomeric chloroacetanilide is produced. When hydrochloric acid is added, the change starts at once, and proceeds regularly; with sulphuric acid, on the other hand, no transformation occurs at first, but after a short interval it begins and proceeds at a constantly increasing rate. At any period, however, during which isomeric change is taking place, hydrogen chloride can be recognised in the solution. The results of the authors confirm the observation first made by Armstrong (*loc. cit.*), that hydrochloric acid has a specific action in the transformation of phenylchloramines.

The experiments were carried out mainly with acetylchloroamino-*p*-chlorobenzene,  $\text{C}_6\text{H}_4\text{Cl}\cdot\text{NCl}\cdot\text{Ac}$ , not only because this substance is obtained somewhat more easily in a pure state, but especially because it changes more slowly into the isomeric anilide than does acetylchloroaminobenzene.

The value of the velocity coefficient, calculated from the equation  $k = 1/t \log a/(a-x)$ , increases with the amount of the catalytic agent and with the temperature, but decreases when the proportion of water in the solvent is increased. In any given experiment, it also begins to increase at some point in the course of the reaction, usually after the first 50 per cent of the chloramine has changed.

The following experiment shows the kind of results obtained:—A 1 per cent solution of acetylchloroamino-*p*-chlorobenzene in glacial acetic acid containing 0.023 per cent hydrogen chloride was kept at a temperature of  $16\cdot5^{\circ}$ , and the unchanged chloramine estimated at the end of one, two, four, six, eight, twenty-four, and twenty-eight hours. Calculating the velocity coefficient from the above equation, the values obtained are respectively 0.0100, 0.0104, 0.0106, 0.0105, 0.0107, 0.0109, and 0.0110; the mean value is 0.0106.

In a series of experiments carried out in order to ascertain the effect of temperature and of variation in the percentage of hydrogen chloride present, and in the concentration of the acetic acid, the following results were obtained:—Using a 1 per cent solution in glacial acetic acid containing 0.023 per cent hydrogen chloride, half the chloramine was transformed in 27.4 hours; but in the presence of 0.069 per cent hydrogen chloride, half was transformed in 5.33 hours, the temperature in each case being  $16\cdot5^{\circ}$ . With a 1 per cent solution containing 0.023 per cent hydrogen chloride, at a temperature of  $35\cdot9^{\circ}$ , half was transformed in 2.75 hours; the value of the velocity-coefficient in this case was not constant, but rapidly increased. Using 50 per cent acetic acid containing 0.023 per cent hydrogen chloride as the solvent, 120 hours elapsed before half was transformed at a temperature of  $16\cdot5^{\circ}$ .

With 0.9 per cent sulphuric acid, instead of hydrogen chloride, in glacial acetic acid, at a temperature of  $16\cdot5^{\circ}$ , no appreciable change occurred in two hours; but after twenty-four hours 8.9 per cent, and after eighty-four hours 50 per cent had changed. Hydrochloric acid, which could not be recognised in the solution after two hours, was found to be present after twenty-four hours.

Similar experiments made with acetylchloroaminobenzene,  $\text{PhNCl}\cdot\text{Ac}$ , show that the isomeric change takes place at a greater rate when the chlorine can wander into the para-position than when it can only pass into the ortho-positions. Thus, in an experiment similar to those just described ( $\text{HCl} = 0.023$  per cent), the time required for the transformation of half the chloramine was two hours instead of 27.4 hours.

In connection with the transformation of phenylacetylchloramines under the influence of hydrogen chloride, the following facts are worthy of note:—When kept in the dark at the ordinary temperature, the titer of a solution in

glacial acetic acid of acetylchloroamino-2:4-dichlorobenzene, which under these conditions cannot be transformed into the isomeric *s*-trichloroacetanilide, slowly falls, and at the same time a vapour (probably hypochlorous acid) is given off, which sets free iodine from potassium iodide. (A similar vapour is evolved from solution of any chloramine in glacial acetic acid). If small quantities of hydrogen chloride or sulphuric acid be present, the titer decreases more rapidly; thus with a solution of the last-mentioned chloramine containing 0.069 per cent of hydrogen chloride, the titer fell 23 per cent at 16.5° in eight days, whilst in the presence of 0.36 per cent sulphuric acid the titer fell 8 per cent of the initial amount in the same period. In both cases the solutions, which were kept in the dark, acquired a yellow colour. Possibly these changes are due to the hydrolysis of the chloramine; the acetic acid contained 1.8 per cent of water. Further, when a solution of hydrogen chloride in dry petroleum is added to a solution of acetylchloroamino-*p*-chlorobenzene in the same solvent, *p*-chloroacetanilide, which is insoluble in petroleum, immediately separates.

Blankens (loc. cit.) has drawn attention to the action of light in effecting this isomeric change, and has observed the transformation of the solid substances under its influence. On exposing any chloramine dissolved in chloroform or in acetic acid to sunlight, the originally colourless solution in a few minutes becomes yellow, and acquires a chlorous smell; in all cases the solution then gives a precipitate with silver nitrate. If a phenylacetylchloramine, which is capable of isomeric change, is so treated, transformation follows. Thus after two hours' exposure to sunlight, acetylchloroamino-*p*-chlorobenzene is changed into the isomeric anilide to the extent of 31 per cent when dissolved in chloroform, but only to the extent of 12 per cent when dissolved in acetic acid. The light is probably active by virtue of the fact that it induces decomposition, in the course of which the catalytic agent is formed.

These results show that in the presence of hydrogen chloride the transformation of phenylacetylchloramines into chloroanilide is *apparently* a monomolecular reaction, and in this way resembles other similar isomeric changes; but it must not be forgotten that in measuring the speed of a chemical reaction the velocity of the slowest of the series of simple changes which constitute the complete transformation is alone measured.

## NOTICES OF BOOKS.

*The Analysis of Steel Works Materials.* By HARRY BREARLEY and FRED. IBBOTSON. London, New York, and Bombay: Longmans, Green, and Co. 1902.

THE importance of a book of this type, which represents the scientific knowledge and experience of thoroughly practical men, can hardly be over-estimated. Too often we have brought before our notice books in which either the practical applications and details are excellent, while the scientific explanations are slovenly or glaringly inaccurate; or the reverse holds good, and methods are advocated which, though excellent from the point of view of a difficult exercise in pure science, are utterly impracticable and absurd from a practical point of view, when the time occupied by the processes, and the intricacy and elaborateness of the apparatus necessary for them are taken into consideration. Such details as economy of time, space, and apparatus are of very great importance in works' laboratories, and have evidently been kept well before the minds of the authors. Undoubtedly, in some cases the complexion of the book reflects, and that very vividly, "personal prejudices." At the same time, it is to be remembered that the prejudices of such authorities as the authors are in all probability well grounded, and less experienced men will gladly avail themselves of the knowledge of those who have had unique opportunities of

acquiring experience and skill. Before passing to a detailed description of the contents of the book, we must relieve ourselves of a word of complaint as to the form into which some of the statements are thrown; a little care would have sufficed to polish some of the sentences, and would have infinitely improved the general style, a by no means insignificant object even in the region of science and laboratories. Careful proof-reading should not have passed by many errors in grammar and orthography which disfigure some pages.

The book is divided into thirteen parts. The first deals with the estimation of all ordinary constituents of steel. The methods described for the determination of carbon do not include the Eggertz colour test, which the authors regard as an accurate method "only under ideal conditions." This is a valuable expression of opinion coming from such a source, since this method has generally been regarded as trustworthy and exact. Methods of distinguishing or estimating the four different modifications of carbon recognised by Ledebur are given from his "Leitfaden für Eisenhütten-Laboratorien." In the case of sulphur, Ledebur's bromine process is apparently considered to be superseded, but a useful and conveniently quick volumetric method is described in detail. Naturally, a great number of methods of estimation of phosphorus are tabulated and described; we may point out that in this as in some other cases the authors appear to somewhat under-estimate the time some of the processes would take; for instance, thirty phosphorus estimations in one day by the method specified appears to be an enormous number, though it is specially stated that this is a task which could not be regularly discharged by one person. In the case of nickel, the authors do not advocate the employment of electrolytic methods of separation and determination, the usefulness and convenience of which they think are, generally speaking, very much over-rated; this opinion we cannot help thinking they will sooner or later see reason to modify. A couple of pages are next devoted to such few methods for the analysis of pig-irons as are not generally employed in steel analysis. Part III. deals with the steel-making alloys; in the case of silicon, special methods are given for the recently introduced high-grade alloys. The determinations of these alloys are most thorough and explicit in all cases, and useful typical results of analyses are added. Short accounts of methods of rapid analysis at the furnace are next given, *i.e.*, processes which are fairly accurate, and in most cases only occupy a few minutes. Such methods of course only aim at approximate results, but those advocated by the authors have certainly the merit of great rapidity; in some cases the time each operation takes is mentioned, and appears to be well within the limits of any possible requirements. Part V. treats of the analysis of iron, manganese, tungsten, and chromium ores, somewhat briefly described. Parts VI. and VII. are devoted to the analysis of various refractory materials, such as ganister, silica bricks, &c., and a few special methods for slags. Fuel is somewhat sketchily treated, the methods of gas analysis being only very briefly indicated. A further development of this section would enhance the usefulness of the book, and render a future edition more complete. Part IX. deals with boiler-water, boiler-scales, &c. Clarke's process for the determination of the hardness of water, although described, is mentioned as rapidly and deservedly growing obsolete, Hehner's method being regarded as more generally useful. For the prevention of scale formation the determination of the necessary amounts of precipitants used is discussed at some length; Kalmann's paper on Bérenger and Stingl's method of softening water being abstracted. In Part X. the analysis of engineering alloys is treated more fully than is usual in books of this kind; the methods given are invariably as simple as possible, and typical examples are always employed, so that by the help of this section the analyst should be able to cope with almost any alloy that may come in his way. An excellent section on the micro-

graphic analysis of steel will undoubtedly be exceedingly useful, instructions for the preparation of samples being fully given, and valuable illustrations of various steels reproduced; these are mostly prepared from specimens belonging to the Metallurgical Department of University College, Sheffield. In connection with this part of the subject, the micro-structure of hardened steel receives special attention, the opinions expressed being confessedly at variance with those generally entertained; the authors' views must of course be judged on their own merits; at any rate they are clearly expressed, and are the results of personal observations and experiments. A short chapter on "Pyrometry" has been specially written for the book by Mr. A. McWilliam, A.R.S.M. Short miscellaneous notes on various subjects embody many practical hints regarding details of manipulation, &c.; these include an abstract of a paper by Franz Hundeshagen on "Phosphododecamolybdic Acid, the Conditions of its Formation, and its Separation as an Ammonium Salt."

A lengthy appendix gives a bibliography of steel-works' analysis, compiled by one of the authors. Various parts of this have already been published in the *CHEMICAL NEWS*; these are now collected and brought up to the end of the year 1901, making a most useful addition to the volume.

*The Electro-plating and Electro-refining of Metals.* Being a New Edition of Alexander Watts's "Electro-deposition." Revised and largely Re-written by ARNOLD PHILIP, A.R.S.M., B.Sc., &c. With numerous illustrations. London: Crosby Lockwood and Son. 1902. Pp. 680.

THIS new edition of Alexander Watts's book on Electro-deposition, appears under a new title, and is divided into two principal parts: Part I. "On Electro-plating," and Part II. "On Electro-metallurgy." It would appear, however, that even this latter sub-title requires modification, inasmuch as the whole of the subject of the electrical extraction of metals from their ores—with the exception of the electrolytic smelting of aluminium—has been excluded from the book, principally, as the author tells us, on account of the unsatisfactory state in which this portion of electro-metallurgical technology still exists.

One of the first impressions given by this volume is its great scope; the amount of matter included is very large, as may be judged from the fact that Part I. consists of twenty-eight chapters and an appendix in two parts, and Part II. contains eight chapters and fourteen useful tables.

After a few chapters on primary and secondary batteries, thermopiles, dynamos, and electro-plating installations, the author gives a short historical review of electro-deposition, followed by five chapters on the practical electro-deposition of copper, in which this important subject is dealt with in a thorough and masterly manner.

Then follow chapters on the electro-deposition of gold, mercury, silver, nickel, tin, and other metals, including many alloys.

Part II. commences with a historical review of the electro-metallurgy of copper; Chapters II. and III. deal with the cost of electrolytic copper refining, current density as a factor in profits, and some important details in electrolytic copper refineries. These two chapters are of an eminently practical nature, and should be carefully studied by copper refiners.

There are several chapters on the electro-refining of other metals, such as gold, silver, lead, nickel, &c.; while Chapter VIII. is on electro-galvanising, or the electro-deposition of zinc on iron, which is the only successfully practised process for the electrolytic treatment of zinc.

The index has been carefully compiled, and is divided into two sections, viz., a subject index and an index of names.

*Seventh Annual Report (for 1900) on Field Experiments on the Manuring of Vegetable and Fruit Crops.* Carried out with the Co-operation of Mr. F. W. E. SHRIVELL at Golden Green, Hadlow, Tonbridge. By Dr. BERNARD DYER. London: Vinton and Co., Ltd. 1902. Pp. 88.

THE seventh experimental year, 1900, on the whole, was a successful one. The experiments on various vegetables and fruits were continued, the latter ones having assumed important dimensions; besides these, an experimental apple orchard has been established, of one acre in extent, divided into six plots, each of which contains a number of varieties of apples.

As in the last report, Dr. Dyer gives under each crop, not only the results of the experiments for the year under consideration, but also a general summary of the collective results of the experience gained with such crop during the seven years of the experimental work.

As has been stated in previous reports, the soil of the main experimental field is a poor clay loam, resting on a deep bed of heavy clay, and is therefore an excellent soil for the purposes of experiment; the result of the spade culture and of assiduous manuring has been to convert the field into a fertile market garden.

The results of seven years' experience has been, to put it briefly, that in nearly every case the advantages obtained by heavy dressings of dung, as so often used, can be obtained sufficiently well and with greater economy by the use of smaller dressings of dung, supplemented with a sufficient quantity of chemical fertiliser to supply a proper quantity of plant food. Full details of the experiments will be found in the pamphlet.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxxv., No. 19, November 10, 1902.

**Artificial Preparation of Rubies by means of Fusion.**—A. Verneuil.—Up to the present time the preparation of artificial rubies has not been successful. The author finds that certain conditions have to be fulfilled in order to prepare aluminium in a transparent form. (1) The melted product must be kept in exactly the same region of the flame; (2) the materials must be packed in thin layers alternately; (3) the points of contact between the melted product and the support should be reduced to as small an area as possible. When these conditions are fulfilled rubies are prepared of a magnificent red fluorescence, of density 4.01. These are found to have the same hardness as natural rubies and to take a good polish. There are, however, often defects in their structure which show their artificial origin, one of these being the presence of different coloured zones due to the decolouration of certain portions.

**Alloys of Copper and Magnesium.**—O. Boudouard.—Alloys of copper and magnesium keep their white colour more or less brilliant until the copper attains a percentage of 70, when the alloys take a faint yellowish tinge. An 80 per cent alloy is yellowish and a 90 per cent one deep yellow. An alloy of 10 per cent copper is malleable; above this it becomes brittle and this property increases until a 70 per cent alloy is reached. Such a substance can then be broken with the fingers. The fragility after this decreases until pure copper is attained. The author, in a series of experiments, compares the various properties of these alloys of magnesium with the corresponding ones of aluminium.

**The presence of Volemite in certain Primulas.**—J. Bougault and G. Allard.—The authors isolated a com-

pound from *Primula grandiflora* which is crystalline and has the properties of a polyatomic alcohol; to this they gave the name of primulite. Further examination, however, shows that it can be identified with volemite, the hepatic alcohol which Bourquelot discovered in *Lactarius volemus*. The authors now describe the method by which this product may be isolated, and investigate its properties. Its empirical formula is  $C_7H_{16}O_7$ .

**Chemical Constitution of Copals.**—Marcel Guérdras. —The author's investigations are carried out on the oil obtained during the pyrogeneration of copals in order to render them soluble for the manufacture of varnish. Three varieties of gum are used:—(1) Copal of Madagascar; (2) copal of Zanzibar; (3) copal of Kausi. He finds in all cases that the purer the copal the larger the quantity of acid. The oils are soluble in alcohol, ether, benzene, and carbon disulphide, but insoluble in the terebenic carbides. When they are treated with nitric acid, a yellow resin is obtained soluble in the solvents mentioned above and also in vegetable oils. They have not, however, succeeded in isolating either cinnamic or benzoic acid or their nitrated derivatives from these oils. The characteristic odour of terpene during the distillation of the oil when oxidised with nitric acid, and the presence of oily globules with an odour of camphor are probably due to the formation of mono-chlorhydrate of terebenthene,  $C_{10}H_{16}HCl$ , which leads the author to suppose that the copals are in part constituted by the terpenes in certain degrees of oxidation.

## MISCELLANEOUS

**Royal Institution.**—A General Monthly Meeting of the Members of the Royal Institution was held on the 1st inst., Sir James Crichton-Brown, Treasurer and Vice-President, in the Chair. The following were elected Members:—Edward Divers, M.D., Miss Amy French, Winifred, Lady Howard of Glossop, and Mr. J. H. Whitehorn. The special thanks of the Members were returned to Mrs. Hickman for her donation of £21, and to Dr. Frank McClean, F.R.S., for his donation of £40 to the Fund for the Promotion of Experimental Research at Low Temperatures.

## MEETINGS FOR THE WEEK.

- MONDAY, 8th.**—Society of Arts, 8. (Cantor Lectures). "The Future of Coal-gas and Allied Illuminants," by Prof. Vivian B. Lewes.
- WEDNESDAY, 10th.**—Society of Arts, 8. "French Rural Education and its Lessons for England," by Cloudeley Brereton.
- THURSDAY, 11th.**—Society of Arts, 4.30. "Domestic Life in Persia," by Miss Ella C. Sykes.
- FRIDAY, 12th.**—Physical, 5. "A Portable Capillary Electrometer," by S. W. J. Smith. "On Astigmatic Aberration," by R. J. Sower. "Experiments on Shadows in an Astigmatic Beam," by Prof. S. P. Thompson. "A Lecture Experiment on Gaseous Diffusion," by Prof. L. R. Wilberforce. "Vapour-density Determinations," by Prof. Sir W. Ramsay, F.R.S., and Dr. B. B. Steele.

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# PHYSIOLOGICAL HISTOLOGY: METHODS AND THEORY

BY

GUSTAV MANN,

M.D., C.M. EDIN.; B.SC. OXON.;

Senior Demonstrator of Physiology, University of Oxford.

**CONTENTS.**—Definition of Histology.—Discussion as to Value of Fixation.—Physical Chemistry.—Colloids and Coagulation.—Chemistry of Reagents used in Fixation.—Formulae of the more important Fixing Fluids.—The Macroscopical Effects of Fixing Reagents.—Microscopical Phenomena of Coagulation and Precipitation.—Guide to the Selection of a Fixative.—Application of Fixatives to Tissues.—On Bleaching, Isolating, and Decalcifying.—On Injecting blood and Lymph Vessels.—Methods of obtaining Sections.—Microtomes.—General Remarks on the Nature of Dyes.—The History of Staining.—Classification of Staining Methods.—Physical Methods of Staining.—Chemical Methods of Staining.—Chemical Methods. Staining after Mordanting.—Staining after Impregnation with Proteid and other Substances.—Impregnation Methods.—On the Chemistry of some Tissue Constituents.—Micro-chemical Reactions.—The Theory of Staining.—How to render Preparations Permanent. **APPENDIX:**—The Chemistry of Dyes. **ADDITIONAL NOTES:**—I. Micro-anatomical Reactions.—II. On Staining.—Electrical Measures.—Tables of Measures and Weights.—Measures of Temperature. **INDEX.**

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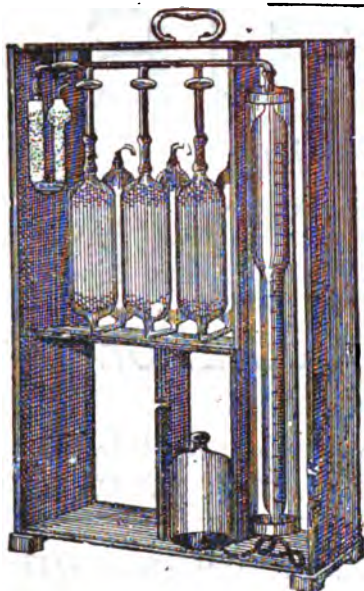
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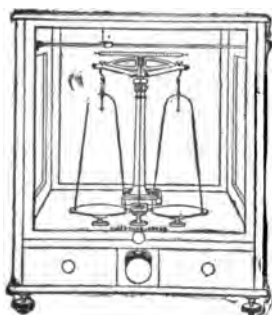
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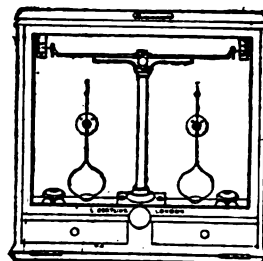


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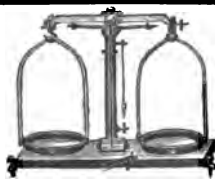
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# THE CHEMICAL NEWS.

VOL. LXXXVI., No. 2246.

## INFUSORIAL EARTH IN IRELAND.

By Dr. T. L. PHIPSON.

A DIATOMACEOUS deposit of considerable extent has been discovered in County Antrim, Ireland, and a specimen was recently sent to me for analysis; the owners of the property on which it was found thinking it was a kind of clay or kaolin.

The microscopic examination at once revealed its real nature, and my analysis gave the following results:—

|                                                 |        |
|-------------------------------------------------|--------|
| Water .. .. .                                   | 10.00  |
| Organic matter and combined water ..            | 5.02   |
| Oxide of iron and alumina .. .                  | 4.99   |
| Lime, magnesia, and alkalis (chiefly lime) .. . | 1.20   |
| Silica .. .                                     | 78.79  |
|                                                 | 100.00 |

When moist it is of a light brown colour, but when dried it is almost quite white. The organic matter shows crenic or apocrenic acid.

The diatoms in this earth are remarkably large and beautiful.

In former years I have noticed that flour is sometimes adulterated with this substance on the Continent, and as this fraud is so easily detected I trust that our Government chemists and others will prevent such flour being used in Great Britain.

Casa Mia Laboratory, Putney,  
December 4, 1902.

## MODIFICATION OF THE RAPID ESTIMATION OF MANGANESE IN STEEL.

By CHARLES RAMORINO.

THE rapid methods employed at the present time in steel-works for the estimation of manganese in steel are nearly all volumetric, and are modified according to the well-known method devised by Volhard, which gives very precise and satisfactory results.

But the manipulation which has to be undertaken specially with a view to the elimination of the reducing carbonaceous matter, and to oxidise the manganese thoroughly, are very long as a rule, and only a very limited number of analyses can be finished in a day, as each one occupies a good deal of time. The modification I propose is the following one; I have applied it in my laboratory, and the results obtained agree perfectly with those obtained by the other methods in general use:—

Two grms. of the sample of steel, taken by means of a drilling machine, are attacked with 30 c.c. of nitric acid; density, 1.20. The attack takes place suddenly in the cold; the metal is allowed to dissolve until we notice flocculent portions of carbon mixed with iron floating about the surface. The solution is completed by heating over a Bunsen burner, and boiling for some minutes until the fumes of the hyponitrite are completely driven off. Then we carefully pour in 20 c.c. of bromised hydrochloric acid, prepared by mixing 10 c.c. of strong hydrochloric acid, density 1.19, and 10 c.c. of strong bromine water. This is boiled, keeping the beaker covered over with a funnel, and kept at the boiling-point for ten minutes until the expulsion of the bromine fumes is complete. We then dilute the solution with about 200 c.c. of cold distilled

water. The whole is then poured into a flask of one litre capacity, and we add 200 c.c. of boiling distilled water, and 25 grms. of pure precipitated oxide of zinc. This is allowed to settle and titrated permanganate is run in from a burette until the rose-colour is persistent.

Several estimations can be made in from fifteen to twenty minutes.

It is necessary to pay attention to the purity of the oxide of zinc used; the ordinary correction for 25 grms. of oxide may vary in a titration where 1 c.c. = 0.00119 Mn from a half to 1 c.c.—*Moniteur Scientifique*, Series 4, vol. xvi., p. 419.

## NOTE ON ISATIN.

By H. J. P. FIRMIN.

WHILE recently preparing a quantity of isatin from indigo by oxidation with nitric acid I made the following observation on separating the resin:—

Most of the resin is precipitated from a strong ammoniacal solution of the resinous and crystalline mass by the cautious addition of diluted hydrochloric acid; the solution filtered, and more acid added till the precipitate shows signs of isatin.

At this point a minute quantity of ammonia is added—with diligent stirring—just sufficient to remove the tendency to a reddish tinge in the precipitate, and the liquid set aside for several days. The resin will precipitate completely without any loss of isatin, and on filtering and adding more acid a most brilliant red precipitate of the latter will occur quite free from resin.

If attempt is made to precipitate all the resin without allowing the solution to stand, either some isatin will come down with the resin, or some of the latter remain with the isatin.

Though in the original mass of resin and crude isatin the former dissolves in ammonia more readily than the latter, yet on precipitating from this solution the resin and some of the isatin with hydrochloric acid, and then, without filtering, adding ammonia, the isatin appears to be taken up before the resin.

It is naturally impossible on paper to describe the exact point at which to add or stop adding the acid or ammonia, but in practice it is soon hit upon.

If a little too much ammonia be added, a trace of acid will put matters right, and *vice versa*.

## A CONTRIBUTION TO THE ELECTRO-CHEMISTRY OF BARIUM COMPOUNDS.\*

By MAX M. HAFF.

THE recent successful extension of electro-chemical methods to the industrial manufacture of the barium compounds, such as the electric furnace method used by the United Barium Company, has led to renewed interest in the details of such manufacture. For a description of the above-mentioned process I would refer to the paper of Mr. Chas. B. Jacobs, in the *Transactions of the American Inst. of Electrical Engineers* for February 28, 1902. Since the separation of the crystalline barium hydrate in this process, by cooling the solution, is of capital importance to the method of manufacture, I have thought that some carefully determined data as to the freezing-points of such solutions and their specific gravity for differing percentages of hydrate,—as obtained in the course of our laboratory work and practice at Niagara Falls, will be of interest to electro-chemists who are following the developments of this process.

\* A Paper read at the Second Meeting of the American Electro-chemical Society, Niagara Falls, September 18, 1902.

Strength of  $\text{BaH}_2\text{O}_2$  Solutions at  $80^\circ \text{C}$ . By Volume and Weight.

| Specific gravity. | Per cent $\text{BaH}_2\text{O}_2$ .<br>Volume. | Per cent $\text{BaH}_2\text{O}_2$ .<br>Weight. |
|-------------------|------------------------------------------------|------------------------------------------------|
| 1'514             | 58'22                                          | 38'45                                          |
| 1'500             | 56'31                                          | 37'54                                          |
| 1'479             | 54'14                                          | 36'60                                          |
| 1'458             | 49'38                                          | 33'87                                          |
| 1'450             | 48'90                                          | 33'72                                          |
| 1'413             | 45'99                                          | 32'55                                          |
| 1'400             | 45'00                                          | 32'14                                          |
| 1'390             | 44'22                                          | 31'81                                          |
| 1'375             | 42'40                                          | 30'84                                          |
| 1'368             | 41'45                                          | 30'30                                          |
| 1'350             | 38'60                                          | 28'59                                          |
| 1'338             | 37'30                                          | 27'88                                          |
| 1'312             | 35'03                                          | 26'69                                          |
| 1'301             | 34'02                                          | 26'13                                          |
| 1'278             | 31'48                                          | 24'67                                          |
| 1'249             | 28'14                                          | 22'52                                          |
| 1'236             | 26'41                                          | 21'36                                          |
| 1'219             | 24'53                                          | 20'12                                          |
| 1'200             | 23'00                                          | 19'17                                          |
| 1'195             | 22'15                                          | 18'53                                          |
| 1'174             | 19'83                                          | 16'89                                          |
| 1'152             | 17'78                                          | 15'43                                          |
| 1'129             | 16'01                                          | 14'18                                          |
| 1'125             | 15'80                                          | 14'04                                          |
| 1'114             | 14'56                                          | 13'07                                          |
| 1'100             | 13'06                                          | 11'87                                          |
| 1'076             | 10'58                                          | 9'83                                           |
| 1'062             | 9'16                                           | 8'62                                           |
| 1'049             | 7'55                                           | 7'20                                           |
| 1'040             | 6'51                                           | 6'26                                           |
| 1'031             | 5'18                                           | 5'02                                           |
| 1'022             | 4'78                                           | 4'67                                           |
| 1'015             | 3'90                                           | 3'84                                           |
| 1'009             | 3'37                                           | 3'34                                           |

Freezing-point of  $\text{BaH}_2\text{O}_2$  Solutions.

| Per cent $\text{BaH}_2\text{O}_2$ . | $^\circ \text{C}$ . |
|-------------------------------------|---------------------|
| 54'23                               | 80'0                |
| 46'7                                | 78'0                |
| 40'7                                | 76'0                |
| 32'7                                | 73'0                |
| 31'3                                | 72'2                |
| 28'6                                | 70'3                |
| 27'1                                | 69'2                |
| 25'6                                | 68'1                |
| 24'0                                | 66'7                |
| 22'4                                | 65'3                |
| 20'6                                | 63'4                |
| 18'4                                | 60'6                |
| 16'9                                | 58'8                |
| 14'8                                | 55'0                |
| 12'0                                | 47'6                |
| 10'4                                | 42'7                |
| 8'2                                 | 34'5                |
| 7'0                                 | 30'0                |
| 4'8                                 | 20'0                |
| 3'0                                 | 9'5                 |
| 2'25                                | 5'0 (a)             |
| 1'75                                | 0'0 (b)             |

- (a) Saturated solution at  $5^\circ \text{C}$ . contains 2'1 per cent.  
 (b) Saturated solution at  $0^\circ \text{C}$ . contains 1'6 per cent.

**The Photogram.**—We have received from the Editors of the *Photogram* a set of cards containing handy memoranda, chiefly upon photographic matters, including guide to correct exposure, rules for telephoto lens, how to find due south without a compass, photographic temperatures, cultivation of stereoscopic vision, &c. The cards are of a convenient size to fit the pocket-book, and the set can be obtained by applying to the office of the *Photogram*, London.

## THE PASTY CONDITION ASSUMED BY ALUMINIUM NEAR ITS POINT OF FUSION, AND THE APPLICATION OF THIS PROPERTY TO THE CUTTING OF THE METAL.

By ALBERT GRANGER.

At the ordinary temperature aluminium is a hard metal. When we wish to cut or divide it by means of the ordinary tools found in a laboratory, such as a saw or a pair of scissors, we encounter a considerable amount of difficulty owing to the toughness of the metal.

When heated to about  $600^\circ$  the metal undergoes a change of structure, and its properties of tenacity and hardness are modified considerably. It becomes granular, and in this condition it can be broken with ease; if the temperature be raised to a sufficiently high point the metal can be completely crushed; it has become quite pasty.

The observation of this phenomenon certainly ought to have been made in the early history of the metallurgy of this metal, but it is singular that no mention is made of it in any of the books dealing with the physical properties of the metal, at least in none that I have come across. My reason for drawing attention to this interesting property, that is met with to a more or less developed extent in other metals, is that it is susceptible of practical application.

Aluminium is often met with commercially in sheets of such a size as to prevent their introduction into the ordinary glass vessels used in laboratories.

These sheets can be cut up very easily, as small as may be wished, by following the directions given below.

The piece of metal is heated (in a muffle furnace for preference) until it is at a dull red heat. From this point onwards aluminium offers much less resistance than at the ordinary temperature. The temperature is allowed to rise until the aluminium can be cut with the blade of an ordinary knife by exerting a little pressure. It can then be cut in strips or slices. The cut surface has a granular appearance, more favourable to attack by reagents than the usual smooth surface it has when cold. By using small quantities at a time it can be powdered in a mortar like zinc. If too large a quantity is tried at a time, the grains become agglomerated owing to the pressure of the pestle.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 15.

**Manganate and Manganite of Barium.**—G. Kassner and H. Keller.—The preparation of manganate of potash cannot be carried out easily or with advantage if we adhere to the conditions demanded by the equation given by Jolles:  $2\text{KMnO}_4 + 2\text{KOH} = \text{H}_2\text{O} + 2\text{K}_2\text{MnO}_4 + \text{O}$ . A larger quantity of potash (KOH) is required, about one and a-half times to twice as much, to prevent the decomposition of the manganate formed. To obtain  $\text{BaMnO}_4$ , we treat  $\text{K}_2\text{MnO}_4$  with chloride of barium. The manganate of barium precipitated contains a little  $\text{BaCO}_3$ , oxides of manganese, and always also a little permanganate which is formed towards the end of the reaction. The molecule of  $\text{BaMnO}_4$  is not fixed to one molecule of water of crystallisation; by drying the salt *in vacuo*, the authors have been able, in fact, to reach such a point that it contained no more than 1'14 per cent of water, while the formula  $\text{BaMnO}_4 \cdot \text{H}_2\text{O}$  requires 6'57 per cent. The reduction of the manganate to the condition of manganite is effected better by means of peroxide of hydrogen than by ferrocyanide of potassium. In fact, this latter salt easily leaves iron in the preparation; washings for the purpose of removing the metal dissociates the manganite into  $\text{Ba(OH)}_2$  and  $\text{MnO}_2$ . The composition of the manganite corresponds to the formula  $\text{BaMnO}_3 \cdot \text{H}_2\text{O}$ ; a desiccation lasting twelve weeks left 6'8 per cent of water in the salt, the preceding formula requiring 6'9 per cent.—*Arch. de Pharm.*, vol. ccxxxix., p. 473.

# CONTRIBUTIONS TO OUR KNOWLEDGE OF YTTERBIUM.\*

By ASTRID CLEVE.

(Continued from p. 277).

## III. COMPOUNDS OF YTTERBIUM (continued).

### Ytterbium Selenates.

#### 1. $\text{Yb}_2\cdot 3\text{SeO}_4 + 15\text{H}_2\text{O}$ .

FROM very concentrated solutions a selenate separates out in the cold in small colourless scales, which probably contain the above amount of water, but possibly crystallise with  $16\text{H}_2\text{O}$ . At  $110^\circ$  the water passes off completely.

#### Analysis of the Pressed Substance.

0.8845 grm. of the substance suffered a loss in weight of 0.2276 grm. at  $110^\circ$ , and after strongly heating gave 0.3389 grm. of  $\text{Yb}_2\text{O}_3$ .

|                            | Calculated for<br>$\text{Yb}_2\cdot 3\text{SeO}_4 + 15\text{H}_2\text{O}$ ,<br>in per cent. | Found. |
|----------------------------|---------------------------------------------------------------------------------------------|--------|
| $\text{Yb}_2\text{O}_3$ .. | 394.2 37.69                                                                                 | 37.19  |
| $3\text{SeO}_3$ ..         | 381.3 36.46                                                                                 | —      |
| $15\text{H}_2\text{O}$ ..  | 270.3 25.85                                                                                 | 25.73  |
|                            | 1045.8 100.00                                                                               |        |

#### 2. $\text{Yb}_2\cdot 3\text{SeO}_4 + 8\text{H}_2\text{O}$ .

Crystallises over the water-bath in large, transparent, hexagonal tables, which crumble quickly in the air. If a solution is cooled while crystallisation is taking place, the formation of this salt ceases, and instead the former salt is obtained, which is stable at lower temperatures.

#### Analysis of the Pressed Salt.

- 0.4618 grm. of the substance lost 0.0723 grm. at  $110^\circ$ , and on strongly heating gave 0.2020 grm.  $\text{Yb}_2\text{O}_3$ .
- 1.3889 grm. (of another preparation) lost 0.2133 grm. at  $120^\circ$ , and on heating gave 0.5914 grm.  $\text{Yb}_2\text{O}_3$ .

|                            | Calculated for<br>$\text{Yb}_2\cdot 3\text{SeO}_4 + 8\text{H}_2\text{O}$ ,<br>in per cent. | Found.        |
|----------------------------|--------------------------------------------------------------------------------------------|---------------|
|                            |                                                                                            | I. II.        |
| $\text{Yb}_2\text{O}_3$ .. | 394.2 42.87                                                                                | (43.73) 42.62 |
| $3\text{SeO}_3$ ..         | 381.3 41.46                                                                                | —             |
| $8\text{H}_2\text{O}$ ..   | 144.16 15.67                                                                               | 15.66 15.43   |
|                            | 919.66 100.00                                                                              |               |

The ytterbium result in Analysis I. is too high, probably owing to the difficulty of expelling the last traces of selenium from the oxide.

#### Specific Gravity and Molecular Volume.

- 1.4075 grm., specific gravity .. .. 3.46
  - 1.4053 " " " .. .. 3.52
- Mean value .. .. 3.49  
Molecular volume .. .. 263

#### 3. Anhydrous Ytterbium Selenate, $\text{Yb}_2\cdot 3\text{SeO}_4$ .

Results on heating either of the previous salts to  $110-120^\circ$ .

Specific Gravity and Molecular Volume.—0.8426 grm. specific gravity, 4.14; molecular volume, 187.

Acid ytterbium selenite,  $\text{Yb}_2\cdot 3\text{SeO}_3 + \text{H}_2\text{SeO}_3 + 4\text{H}_2\text{O}$ , was prepared and analysed by Nilson (*loc. cit.*, p. 10).

### Ytterbium Carbonates.

#### 1. Neutral Ytterbium Carbonate, $\text{Yb}_2\cdot 3\text{CO}_3 + 4\text{H}_2\text{O}$ .

Results as a colourless gelatinous precipitate on precipitation of a neutral solution of ytterbium nitrate with ammonium carbonate; after washing, it is free from ammonia.

#### Analysis of the Salt Dried in the Air.

- 0.7176 grm. of the substance gave up 0.0270 grm. of water over sulphuric acid, and at  $100^\circ$  0.0538 grm. more. After precipitation with oxalic acid and ignition, 0.4771 grm. of  $\text{Yb}_2\text{O}_3$  was obtained.
- 0.5270 grm. of the substance gave 0.3482 grm.  $\text{Yb}_2\text{O}_3$ .

|                            | Calculated for<br>$\text{Yb}_2\cdot 3\text{CO}_3 + 4\text{H}_2\text{O}$ ,<br>in per cent. | Found.      |
|----------------------------|-------------------------------------------------------------------------------------------|-------------|
|                            |                                                                                           | I. II.      |
| $\text{Yb}_2\text{O}_3$ .. | 394.2 65.89                                                                               | 65.80 66.08 |
| $3\text{CO}_2$ ..          | 132. 22.06                                                                                | —           |
| $4\text{H}_2\text{O}$ ..   | 72.08 12.05                                                                               | 11.26 —     |
|                            | 598.28 100.00                                                                             |             |

Three molecules of water were retained over sulphuric acid. In the salt thus dried the percentage of  $\text{Yb}_2\text{O}_3$  found was 68.36 per cent (I.) and calculated for  $\text{Yb}_2\cdot 3\text{CO}_3 + 3\text{H}_2\text{O}$  was 67.94 per cent.

#### Specific Gravity and Molecular Volume.

- 0.8794 grm. of the substance dried over sulphuric acid, specific gravity .. .. 3.676
  - 1.0227 grm. of the substance dried over sulphuric acid, specific gravity .. .. 3.663
- Mean value .. .. 3.670  
Molecular volume .. .. 158

#### 2. Basic Ytterbium Carbonate, $\text{YbOHCO}_3 + \text{H}_2\text{O}$ (Dried over Sulphuric Acid).

If carbon dioxide is passed for some time (about twenty-four hours) through freshly precipitated ytterbium hydroxide suspended in water, the precipitate remains—as far as outward appearance is concerned—unchanged and gelatinous, and also after heating is quite amorphous, but has, as a matter of fact, been converted into the basic carbonate. At  $100^\circ$  the salt loses half the water, which is retained in the desiccator.

#### Analysis of the Salt Washed with Alcohol-ether and Dried over Sulphuric Acid.

- 0.5342 grm. of the substance gave, on heating in a combustion tube, 0.0541 grm.  $\text{H}_2\text{O}$  and 0.3914 grm.  $\text{Yb}_2\text{O}_3$ .
- 0.5030 grm. of the substance lost at  $100^\circ$  0.0283 grm. and gave 0.3676 grm.  $\text{Yb}_2\text{O}_3$ .

|                                     | Calculated for<br>$\text{YbOHCO}_3 + \text{H}_2\text{O}$ ,<br>in per cent. | Found.                              |
|-------------------------------------|----------------------------------------------------------------------------|-------------------------------------|
|                                     |                                                                            | I. II.                              |
| $\text{Yb}_2\text{O}_3$ ..          | 394.2 73.51                                                                | 73.77 73.04                         |
| $2\text{CO}_2$ ..                   | 88. 16.41                                                                  | —                                   |
| $3\text{H}_2\text{O}$ ..            | 54.06 10.08                                                                | 10.13 —                             |
|                                     | 536.26 100.00                                                              |                                     |
| $1\frac{1}{2}\text{H}_2\text{O}$ .. | 5.04                                                                       | 5.62 (loss of wt. at $100^\circ$ ). |

In respect of the existence and method of preparation of the basic carbonate, ytterbium is allied to erbium and gadolinium, which both form under similar conditions salts of the same type (Cleve and Höglund, *Bull. Soc. Chim.*, 1872, xviii., 293; Benedicks, *Zeit. Anorg. Chem.*, xxii., 417). Samarium (Cleve, "Contributions to the Knowledge of Samarium," p. 23) and praseodymium hydroxides (v. Schéele, "Om Praseodym, &c.," *Inaug. Diss.*, Upsala, 1900, p. 66), on the other hand, take up  $\text{CO}_2$  until neutral carbonates are formed.

On precipitating neutral ytterbium solutions with sodium carbonate, gelatinous precipitates are formed, which cannot be completely freed from sodium by washing. Up to the present it has not been definitely ascertained whether the retention of the sodium is due to the formation of a double salt, or to adhesion.

#### Ytterbium Borate, $\text{YbBO}_3$ .

Ytterbium oxide is only with difficulty attacked by boric anhydride when heated before the blowpipe.

\* From the *Zeitschrift für Anorganische Chemie*, xxii.



On heating for several hours with  $B_2O_3$  in great excess, a fused mass is obtained, which, after being broken in pieces, is freed from unchanged boric anhydride by treating with dilute hydrochloric acid, ytterbium ortho-borate being then left behind as a fine-grained crystalline sand. Since concentrated hydrochloric acid has no appreciable effect on the salt, for purposes of analysis it is evaporated with hydrofluoric acid, and the fluoride decomposed by means of sulphuric acid.

#### Analyses.

1. 1.0798 grm. of the substance in the coarsest possible crystals gave 1.4565 grm.  $Yb_2O_3$ .
2. 0.9440 grm. of the substance in fine crystals gave 1.2600 grm.  $Yb_2O_3$ .

|              | Calculated for<br>$YbBO_3$ , in per cent. |        | Found. |       |
|--------------|-------------------------------------------|--------|--------|-------|
|              | I.                                        | II.    | I.     | II.   |
| $Yb_2O_3$ .. | 394.2                                     | 84.92  | 85.77  | 84.87 |
| $B_2O_3$ ..  | 70.                                       | 15.08  | —      | —     |
|              | 464.2                                     | 100.00 |        |       |

Cleve has prepared analogous ortho-borates of didymium and samarium.

#### Ytterbium Phosphates.

##### 1. Ytterbium Ortho-phosphate, $YbPO_4 + 4\frac{1}{2}H_2O$ .

From neutral solutions of the nitrate the ytterbium is completely precipitated by ordinary sodium phosphate ( $Na_2HPO_4$ ). After washing, the gelatinous precipitate exhibits a neutral composition, while the filtrate and washings are rendered acid by phosphoric acid. Dried in the air, the phosphate thus obtained forms heavy semi-transparent particles, which give up some water over sulphuric acid, but even at  $100^\circ$  retain  $2H_2O$ .

#### Analyses of the Salt Dried in the Air.

1. 2.7426 grms. of the substance lost 0.3230 grm. in the desiccator; 1.4289 grms. of the residue gave up 0.0345 grm. of water at  $100^\circ$ .
2. 1.0302 grms. of the substance gave up 0.1334 grm. at  $100^\circ$ , and 0.0992 grm. more water before the blow-pipe.
3. 1.0602 grms. of the substance gave when fused with soda 0.3284 grm.  $Mg_2P_2O_7$  and 0.5620 grm.  $Yb_2O_3$ .
4. 0.5918 grm. of the substance gave 0.1956 grm.  $Mg_2P_2O_7$ .

|              | Calculated for<br>$YbPO_4 + 4\frac{1}{2}H_2O$ ,<br>in per cent. |        | Found. |                                            |                                              |       |
|--------------|-----------------------------------------------------------------|--------|--------|--------------------------------------------|----------------------------------------------|-------|
|              | I.                                                              | II.    | III.   | IV.                                        |                                              |       |
| $Yb_2O_3$ .. | 394.2                                                           | 56.44  | —      | —                                          | 55.91                                        | —     |
| $P_2O_5$ ..  | 142.                                                            | 20.34  | —      | —                                          | 20.81                                        | 21.07 |
| $4H_2O$ ..   | 72.08                                                           | 10.32  | —      | —                                          | 9.63 (loss of weight<br>above $100^\circ$ ). |       |
| $5H_2O$ ..   | 90.10                                                           | 12.90  | 13.89  | 12.95 (loss of weight<br>at $100^\circ$ ). |                                              |       |
|              | 698.38                                                          | 100.00 |        |                                            |                                              |       |

Although the results of the analyses do not agree very well with the theory,—as is scarcely to be expected in the case of amorphous precipitates such as this,—there can be no doubt concerning the neutral composition of this phosphate. Neutral ortho-phosphates of yttrium and erbium are similarly formed (Cleve; Cleve and Höglund).

##### 2. Ytterbium Meta-phosphate, $Yb_3PO_3$ .

Attempts to prepare the phosphate  $Yb_2O_3 \cdot 5P_2O_5$  according to the method described by Cleve, i.e., solution of the anhydrous sulphate in fused meta-phosphoric acid, led to the formation of the meta-phosphate  $Yb_3PO_3$ , and not the desired compound. On boiling with water, the fused mass was for the most part dissolved. Only small quantities of a crystalline sand remained behind, and this was analysed.

1. 0.3287 grm. of the substance gave 0.2735 grm.  $Mg_2P_2O_7$ , and 0.1534 grm.  $Yb_2O_3$ .
2. 0.2718 grm. of the substance (fresh preparation) gave 0.1316 grm.  $Yb_2O_3$ .

|              | Calculated for<br>$Yb_3PO_3$ , in per cent. |        | Found. |       |
|--------------|---------------------------------------------|--------|--------|-------|
|              | I.                                          | II.    | I.     | II.   |
| $Yb_2O_3$ .. | 394.2                                       | 48.03  | 46.67  | 48.42 |
| $3P_2O_5$ .. | 426.                                        | 51.97  | 53.06  | —     |
|              | 820.2                                       | 100.00 | 99.73  |       |

According to the analysis, some phosphoric acid was still mixed with Preparation I.

The corresponding yttrium compound is known.

##### 3. Ytterbium Phosphate, $Yb_2O_3 \cdot 2P_2O_5 + 5H_2O$ .

In the preparation of the last salt most of the ytterbium went into the aqueous solution, as was pointed out. It was recovered from the solution, after strongly concentrating, as a gelatinous salt,  $Yb_2O_3 \cdot 2P_2O_5$ , but could not be separated directly by means of ammonia, probably owing to the formation of colloid soluble phosphates. After some time the solution becomes turbid, and the addition of ammonium chloride at once causes the formation of a precipitate, which again dissolves on washing.

When dried in the air,  $Yb_2O_3 \cdot 2P_2O_5$  forms a heavy powder with 5 molecules of water, three of which are retained at  $100^\circ$ .

#### Analysis of the Salt Dried in Air.

- 0.3263 grm. of the substance lost 0.081 grm. at  $100^\circ$ ; when heated before the blowpipe a further loss of 0.0226 occurred. 0.1854 grm.  $Mg_2P_2O_7$  was obtained by the above method from the residue, and 0.1679 grm.  $Yb_2O_3$  by precipitation with oxalic acid.

|              | Calculated for<br>$Yb_2O_3 \cdot 2P_2O_5 + 5H_2O$ ,<br>in per cent. |       | Found.                                |     |
|--------------|---------------------------------------------------------------------|-------|---------------------------------------|-----|
|              | I.                                                                  | II.   | I.                                    | II. |
| $Yb_2O_3$ .. | 394.2                                                               | 51.30 | 51.46                                 | —   |
| $2P_2O_5$ .. | 284.                                                                | 36.97 | 36.22                                 | —   |
| $3H_2O$ ..   | 54.06                                                               | 7.04  | 6.93 (loss of wt. above $100^\circ$ ) | —   |
| $2H_2O$ ..   | 36.04                                                               | 4.69  | 5.55 (loss of wt. at $100^\circ$ )    | —   |
|              | 768.3                                                               |       |                                       |     |

#### Ytterbium Vanadates.

##### 1. $3Yb_2O_3 \cdot 5V_2O_5 + 3H_2O$ (Dried at $100^\circ$ ).

Obtained as a yellow precipitate from ytterbium nitrate and ammonium vanadate.

#### Analysis.

- 0.5061 grm. of the substance dried at  $100^\circ$  gave, when dissolved in HCl and precipitated with oxalic acid, 0.2766 grm.  $Yb_2O_3$ . By treating with nitric acid, 0.2158 grm.  $V_2O_5$  was obtained from the filtrate.

|               | Calculated for<br>$3Yb_2O_3 \cdot 5V_2O_5 + 3H_2O$ ,<br>in per cent. |        | Found. |     |
|---------------|----------------------------------------------------------------------|--------|--------|-----|
|               | I.                                                                   | II.    | I.     | II. |
| $3Yb_2O_3$ .. | 1182.6                                                               | 55.04  | 54.65  | —   |
| $5V_2O_5$ ..  | 912.                                                                 | 42.45  | 42.64  | —   |
| $3H_2O$ ..    | 54.1                                                                 | 2.51   | —      | —   |
|               | 2148.7                                                               | 100.00 |        |     |

##### 2. $Yb_2O_3 \cdot 15V_2O_5$ .

On concentrating the filtrate from the above insoluble vanadate over sulphuric acid, a brown crust was deposited which, on analysis, gave a considerably higher percentage of vanadium, although ytterbium nitrate was present in excess in the solution.

#### Analysis.

- 0.4112 grm. of the substance analysed as above gave 0.0530 grm.  $Yb_2O_3$  and 0.3582 grm.  $V_2O_5$ .

|               | Calculated for<br>$Yb_2O_3 \cdot 15V_2O_5$ , in per cent. |        | Found. |     |
|---------------|-----------------------------------------------------------|--------|--------|-----|
|               | I.                                                        | II.    | I.     | II. |
| $Yb_2O_3$ ..  | 394.2                                                     | 12.55  | 12.89  | —   |
| $15V_2O_5$ .. | 2536.                                                     | 87.45  | 87.13  | —   |
|               | 2930.2                                                    | 100.00 |        |     |

**Basic Potassium-ytterbium Chromate,**  
 $2KYb_2CrO_4 + Yb(OH)_3 + 15\frac{1}{2}H_2O$ .

If a neutral solution of ytterbium nitrate is mixed with neutral potassium chromate a yellow amorphous precipitate is formed, while potassium bichromate gives no precipitate.

The ytterbium salt must be present in excess in order that this double compound may be formed, otherwise the solution becomes yellowish red, and the precipitate then remains basic. As water causes decomposition, the salt is pressed directly for analysis without washing.

**Analyses.**

- 0.6831 grm. of the substance suffered a loss of weight of 0.1748 grm., equals 25.59 per cent at 100°. For  $11H_2O$  the calculated loss is 24.93 per cent.
- 0.5055 grm. of the salt dried at 100° gave, on addition of  $HgNO_3$ , 0.0658 grm.  $Cr_2O_3$ . After the excess of mercury had been removed by means of sulphuretted hydrogen, 0.3142 grm.  $Cr_2O_3 + Yb_2O_3$  was precipitated with ammonia; 0.2568 grm. of this precipitate was  $Yb_2O_3$ . After the precipitate had been removed by filtration the solution gave 0.0360 grm.  $K_2O$ , weighed as  $KCl$ .

|               | Calculated for<br>$2KYb_2CrO_4 + Yb(OH)_3 + 4H_2O$ ,<br>in per cent. |        | Found. |
|---------------|----------------------------------------------------------------------|--------|--------|
| $3Yb_2O_3$ .. | 1182.6                                                               | 49.52  | 50.74  |
| $2K_2O$ ..    | 188.6                                                                | 7.90   | 7.12   |
| $8CrO_3$ ..   | 800.8                                                                | 33.53  | 32.15  |
| $12H_2O$ ..   | 216.24                                                               | 9.05   | —      |
|               | 2388.24                                                              | 100.00 |        |

In preparing this substance, as was mentioned before, the ytterbium salt was present in excess, and as the salt was not washed, the K and Cr determinations are rather too low, and the Yb, on the other hand, somewhat high.

**Ytterbium Molybdates.**

**1.  $Yb_2O_3 \cdot 7MoO_3 + 6H_2O$  (Dried at 130°).**

A solution of ytterbium nitrate, to which ammonium molybdate,  $3(H_4N)_2O \cdot 7MoO_3$ , has been added in the cold, is not at once rendered turbid, but after some time the above salt separates out as a colourless opaque crust, which is insoluble even in hot water.

**Analysis of the Salt Dried at 130°.**

0.3692 grm. of the substance dissolved in nitric acid gave, on precipitation with ammonia, 0.1059 grm.  $Yb_2O_3$ . 0.2608 grm.  $MoO_3$  was separated from the filtrate by  $HgNO_3$ .

|              | Calculated for<br>$Yb_2O_3 \cdot 7MoO_3 + 6H_2O$ ,<br>in per cent. |        | Found. |
|--------------|--------------------------------------------------------------------|--------|--------|
| $Yb_2O_3$ .. | 394.2                                                              | 26.10  | 26.73  |
| $7MoO_3$ ..  | 1008.                                                              | 66.74  | 65.83  |
| $6H_2O$ ..   | 108.17                                                             | 7.16   | —      |
|              | 1510.37                                                            | 100.00 |        |

**2.  $2Yb_2O_3 \cdot MoO_3$ .**

If ytterbium earth is heated with excess of molybdenum trioxide in a flux of common salt, the earth is attacked only with very great difficulty, even when heated strongly before the blowpipe. The greater part remains undissolved, but it is finally converted into a heavy dark green crystalline powder.

**Analysis.**

0.7152 grm. of the substance gave, when treated with soda, 0.1088 grm.  $MoO_3$  and 0.6052 grm.  $Yb_2O_3$ .

|               | Calculated for<br>$2Yb_2O_3 \cdot MoO_3$ , in per cent. |  | Found. |
|---------------|---------------------------------------------------------|--|--------|
| $2Yb_2O_3$ .. | 84.56                                                   |  | 84.62  |
| $MoO_3$ ..    | 15.44                                                   |  | 15.21  |
|               | 100.00                                                  |  | 99.83  |

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, Wednesday, November 19, 1902.

Prof. J. EMERSON REYNOLDS, Sc.D., V.P.R.S., President,  
in the Chair.

CERTIFICATES were read for the first time in favour of Messrs. Gilbert John Alderton, Eglinton Road, Plumstead, S.E.; Harry Percy Lewis, Swansea Hematite Works, Landore; Thomas Samuel, School of Science and Art, Liscard, Cheshire; John Mello Wadmore, B.A., Meriden, Exmouth; Albert Wilmore, 158, Skipton Road, Colne; William Wade Yeomans, Middlewich, Cheshire.

The PRESIDENT announced that the Council that afternoon had appointed Dr. G. T. Morgan to be Editor of the publications of the Society from January 1st next.

Of the following papers, those marked \* were read:—

\*160. "The 'Dynamic Isomerism' of Thiourea and Ammonium Thiocyanate." By J. E. REYNOLDS, D.Sc., F.R.S., and E. A. WERNER, F.I.C.

The method of producing thiourea for use in the arts is still essentially that by which one of the authors succeeded in isolating the substance in 1868 (*Trans.*, vii., 1). This consists in carefully heating fused ammonium thiocyanate so as to secure as far as possible the molecular rearrangement represented by  $NH_4CNS = CSN_2H_4$ .

The proportion of thiourea actually formed at any single operation of this kind is known to be small. Volhard and others have variously estimated the yield at 15 to 22 per cent of the thiocyanate taken for conversion. It is also well known that pure thiourea, when melted, slowly reverts to ammonium thiocyanate; but the estimates of the extent of this reversion also vary considerably. Viewed as a "balanced action" conditioned by heat alone, these changes are cited by Lowry as examples of "dynamic isomerism."

In view of these discrepancies, which they considered were probably due to the use of small quantities of impure or moist materials, the authors have systematically studied on a comparatively large scale the effects of temperature and time on the course of these changes, and give the results of their experiments in curves. They show that:—

1. Ammonium thiocyanate fused, and then raised to, and maintained at, a steady temperature of 170°, underwent gradual change into thiourea until a maximum of 24.7 per cent was reached after forty-five minutes. Continued heating for twenty-five minutes longer led to slight loss of thiourea by decomposition.

2. In another series of experiments, the mean temperature being 182°, similar results were obtained, but the change was more rapid in the earlier stages, although the time required for the maximum effect was substantially the same as before.

3. Pure thiourea fused, and then maintained at a steady temperature of 170°, gradually reverted to ammonium thiocyanate, and after forty minutes only 25.8 per cent of thiourea remained. That proportion was not materially diminished by continued heating.

Therefore equilibrium is reached from either side when the isomerides are present in the melt in proportions which are substantially 25 per cent of thiourea and 75 per cent of ammonium thiocyanate, which is the composition of a definite compound having the formula  $CSN_2H_4 \cdot (NH_4CNS)_3$ .

The authors conclude that the arrest of change is due to the production of this compound, which appears to be more stable at high temperatures than either of its constituents.

In confirmation of this view, they find that crystalline aggregates of nearly the above composition can be separated from the centre of a slowly cooled mass, produced by heating about a kilogram of ammonium thiocyanate to 170° for forty minutes.

Further, having noted that the material which contained about 25 per cent of thiourea remained liquid down to  $106^\circ$ , they determined the points of complete liquefaction of a number of very intimate mixtures of pure thiourea and thiocyanate. They found that there was a regular decrease in melting-point from  $148^\circ$  (that of pure thiocyanate) to  $106^\circ$ , at which latter temperature a mixture containing 25 per cent of thiourea fused, but with still higher percentages of thiourea there was a regular rise in melting-point, as shown on a curve of eutectic form having its minor-point at  $106^\circ$ .

Finally, it was pointed out that the dissociation of the high temperature compound by solvents is by no means complete unless the latter are used in comparatively large proportions. Thus, when the melt was treated with insufficient warm acetone to dissolve it wholly, the solution afforded a compound which afterwards separated in feathery groups of crystals. This substance proved on analysis to be  $CSN_2H_4 \cdot NH_4CNS$ . With alcohol and water, the process of breaking down proceeds farther, but even warm water, if used in small proportion, does not cause complete separation, as fine, long crystals were easily obtained from such a solution which consisted of  $(CSN_2H_4)_3 \cdot NH_4CNS$ .

This compound is obviously complementary in composition to that which is formed in the fused mass at  $170^\circ$ . Re-resolution of the crystals in water leads to complete separation.

Generally, the authors conclude that the state of equilibrium reached when either isomeride is heated to  $170^\circ$  for forty minutes is conditioned as much by perfectly definite chemical attraction as by heat.

\*161. "Isomeric Partially Racemic Salts containing Quinquevalent Nitrogen. Part VIII. Resolution of the Hydrindamine Bromocamphorsulphonates." By F. S. KIPPING.

In previous communications (*Trans.*, 1900, lxxvii., 861; 1901, lxxix., 430), the author has described several cases of a new type of isomerism observed in the study of salts of *dl*-hydrindamine (and of *dl*-benzylhydrindamine) with bromo- and chloro-camphorsulphonic acids and with *cis*- $\pi$ -camphanic acid; in order to account for the existence of these isomerides, it was assumed that each of the optically active bases gives rise to two salts, and that the four compounds thus formed unite in pairs to produce the two partially racemic isomerides.

Further investigation, which so far has been restricted to the isomeric hydrindamine bromocamphorsulphonates (distinguished as the " $\alpha$ "- and " $\beta$ "-salts), seems to show that this view of their nature is substantially correct.

**Resolution of the  $\alpha$ -Salt.**—When the  $\alpha$ -salt (m. p.  $150^\circ$ ), which separates unchanged from most ordinary solvents, is heated with a little ethyl acetate under suitable conditions, it is resolved into two components, one of which is deposited from the hot solution, whilst the other can be isolated from the mother-liquors. The former, provisionally called the  $\alpha A$ -salt, crystallises in needles melting at about  $223^\circ$ ; the latter, the  $\alpha B$ -salt, crystallises in needles melting at about  $130^\circ$ .

These two salts have practically the same specific rotation in dilute aqueous solution, namely,  $[\alpha]_D = +60^\circ$ ; the molecular rotation, therefore, in both cases is  $+266^\circ$ , a value identical with that of bromocamphorsulphonic acid. The two compounds represent, nevertheless, salts of the enantiomorphously related hydrindamines, the bases having such low molecular rotations that they show no appreciable activity, even in moderately concentrated solutions of their hydrochlorides. The hydrochlorides, prepared from the  $\alpha A$ - and  $\alpha B$ -salts, are identical in ordinary properties, but the salts obtained by combining the bases from  $\alpha A$  and  $\alpha B$  with optically active acids are different; it follows, therefore, that the enantiomorphously related bases do not undergo racemisation when liberated from their salts.

**Resolution of the  $\beta$ -Salt.**—The partially racemic  $\beta$ -salt (m. p.  $130^\circ$ ) is also resolved into different components

when it is crystallised from hot ethyl acetate under suitable conditions. The salt, which separates from the hot solution, crystallises in needles which appear homogeneous and which resemble  $\alpha A$  very closely in outward and in optical properties; when, however, this compound is systematically crystallised from water, after some 30–40 operations, it is further resolved into a salt identical with  $\alpha A$  and a new isomeride  $\beta A$ ; the latter is very similar to  $\alpha A$ , but melts at a lower temperature (about  $215^\circ$ ), and has a lower specific rotation ( $[\alpha]_D = +56^\circ$ ) in aqueous solution; it is possible that this salt may not have been obtained quite free from  $\alpha A$ .

The ethyl acetate mother-liquors, from which  $\alpha A$  and  $\beta A$  have been deposited, contain unchanged  $\beta$ -salt together with two isomerides; one of these is identical with  $\alpha B$ , but the other,  $\beta B$ , although very similar to  $\alpha B$  in some properties, can be separated from it by fractional crystallisation from water. This fourth isomeride,  $\beta B$ , melts at about  $130^\circ$ , and has a specific rotation  $[\alpha]_D = +39.5^\circ$  in aqueous solution.

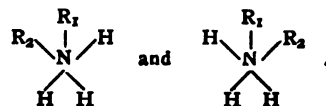
**Synthesis of the  $\alpha$ - and  $\beta$ -Salts.**—When equal quantities of  $\alpha A$  and  $\alpha B$  are dissolved together in hot water, the solution deposits the partially racemic  $\alpha$ -salt, and fractional crystallisation fails to reveal the presence of any  $\beta$ -salt in the product.

When  $\alpha A$  and  $\alpha B$  are separately decomposed with baryta, and the liberated bases are neutralised with the bromo-acid, the salts obtained appear to be identical with  $\alpha A$  and  $\alpha B$  respectively, but they are not so; for if now mixed in equal quantities and crystallised from water, they give a product which consists of a mixture of the  $\alpha$ - and  $\beta$ -partially racemic salts. This is because, on uniting with the acid, the one base gives  $\alpha A$  and  $\beta A$ , the other  $\alpha B$  and  $\beta B$ ; the salt produced by combining the base from  $\alpha B$  with the acid may, in fact, be resolved into  $\alpha B$  and  $\beta B$  by fractional crystallisation.

When equal quantities of  $\beta A$  and  $\beta B$  are dissolved together in water, there results a salt which crystallises in hydrated prisms, indistinguishable in appearance from those of the partially racemic  $\beta$ -salt (m. p.  $130^\circ$ ), but which, when dehydrated, melts at about  $165^\circ$ ; this fact, and the constant occurrence of four compounds in the products of the resolution of samples of the  $\beta$ -salt which have been repeatedly crystallised to free them from  $\alpha$ -salt, show that the  $\beta$ -salt is composed of four isomerides. The salt composed of equal quantities of  $\beta A$  and  $\beta B$  seems to represent a new isomeride, in which case there would be three partially racemic salts.

\*162. "Isomeric Compounds of the Type  $NR_1R_2H_3$ ." By F. S. KIPPING.

From the results summarised in the preceding note, it will be seen that six isomeric salts have now been isolated from the mixture produced by the combination of *dl*-hydrindamine and *d*-bromocamphorsulphonic acid. Four of these salts,  $\alpha A$ ,  $\alpha B$ ,  $\beta A$ , and  $\beta B$  contain one molecule of one of the enantiomorphously related bases combined with one molecule of the acid; that is to say, each of the bases gives rise to two isomerides which may be represented by the symbols:—



Such compounds containing only three different radicles united with quinquevalent nitrogen, afford an example of a type of isomerism hitherto unknown; it is possible, however, that the isomerism of the optically inactive compounds of the type  $NR_1R_2R_3R_4R_5$ , described by Wedekind, may be of an analogous character.

The existence of these new isomerides has no doubt an important bearing on the question of the arrangement in space of the groups around a quinquevalent atom, and would seem to exclude the double tetrahedron configuration; also the view, very generally held, that the negative

ion in an ammonium salt takes up one particular valency. The fact that, in the case examined, the two radicals,  $R_1$  and  $R_2$ , are enantiomorphous does not of course preclude the possibility of the existence of isomerides in which the three different radicals are all optically inactive, and it may even be possible that an apparently simple salt, such as ammonium chloride, is in reality a mixture of two isomerides.

One particularly striking fact in connection with the isomeric hydrindamine salts is their stability; all the four simple isomerides have been repeatedly crystallised from boiling water and other solvents, and hitherto the conversion of one into the other has never been observed.

It is also interesting to note the highly abnormal molecular rotation of the  $\beta\beta$ -isomeride; in 2 per cent aqueous solution,  $[M]_D = +175^\circ$ , whereas that of the acid is  $[M]_D = +270^\circ$ . Since the ordinary base ion, although enantiomorphous, has only an extremely low molecular rotation, it might be inferred that the  $\beta\beta$ -isomeride is only very partially dissociated, and that there are two distinct types of ammonium salts.

\*163. "The Oxime of Mesoxamide and some Allied Compounds. Part II. Disubstituted Derivatives." By Miss M. A. WHITELEY, D.Sc.

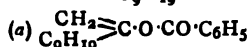
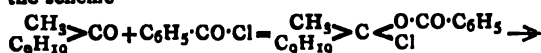
The following mono- and di-substituted derivatives of the oxime of mesoxamide have been prepared:—*isomitosomalondimethylamide*, m. p.  $157^\circ$ , the *potassium* and *ferrous* salts; *isomitosomalonanilide*, m. p.  $141^\circ$ , the *potassium*, *ferrous*, and *silver* salts; *isomitosomalondip-tolylamide*, m. p.  $170-171^\circ$ , the *potassium* and *silver* salts; *isomitosomalonmono-p-tolylamide*, m. p.  $183^\circ$ ; *isomitosomalondio-tolylamide*, m. p.  $111^\circ$ , the *potassium* salt; *ethyl isomitosomalon-o-tolylamide*, m. p.  $140-141^\circ$ ; *isomitosomalondianaphthylamide*, m. p.  $184^\circ$ , the *potassium* salt; *isomitosomalondinaphthylamide*, m. p.  $221^\circ$ , the *acetyl* derivative, m. p.  $179^\circ$ , a chlorinated derivative, m. p.  $202^\circ$ .

All these compounds possess the characteristic salt-forming properties exhibited by the original oxime, the alkali salts being yellow and the ferrous salts purple or blue. An interesting tautomerism is exhibited by some of them, the two isomerides differing from one another in colour (one being yellow and the other colourless), crystalline form, and solubility. The most probable explanation of this tautomerism is the assumption that the colourless isomeride has the *isoximino*-structure, whilst the yellow isomeride and the yellow salts correspond to the *oximino*-compound.

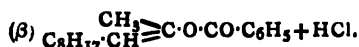
The examination of a fairly extensive series of  $\alpha$ -oximino-ketones has shown that in addition to the well-known characteristic yellow colouration with alkalis, almost all give a deep purple or blue colour on the further addition of ferrous sulphate. This latter characteristic, however, appears to be influenced by the isomerism of the oximino-group, for  $\alpha$ -benzil monoxime gives the reaction, whilst the  $\beta$ -isomeride does not. Again, among the dioximes, a considerable number, including  $\alpha$ -benzil dioxime, were found to give a deep purple or brown colouration with ferrous sulphate and an alkali, whilst  $\beta$ - and  $\gamma$ -benzil dioxime gave no colouration under these conditions.

\*164. "Interaction of Ketones and Aldehydes with Acid Chlorides—the Formation of Benzoxalofines and *1-benzoxycamphens*." By F. H. LEES.

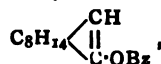
The author has found that methyl *n*-nonylketone and benzoyl chloride readily interact when boiled together, forming  $\beta$ -benzoxynonyldecylene ( $\alpha$  or  $\beta$ ),  $\text{CH}_3\text{C}(\text{OBz})\text{CH}_2\text{C}_8\text{H}_{17}$  or  $\text{CH}_3\text{C}(\text{OBz})\text{CH}\cdot\text{C}_8\text{H}_{17}$ . It is an oil, b. p.  $233-235^\circ$  (50 m.m.), and is regarded as being formed according to the scheme—



or—

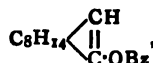


This formation of  $\beta$ -benzoxynonyldecylene represents the first instance of the direct transformation of a mono-oxygenated ketone into a derivative of its enolic form. The author has extended this reaction to other ketones, an aldehyde, and an aliphatic acid chloride, the following substances resulting:— $\beta$ -Benzoxynonyldecylene ( $\alpha$  or  $\beta$ ),  $\text{CH}_3\text{C}(\text{OBz})\text{C}_7\text{H}_{15}$  or  $\text{CH}_3\text{C}(\text{OBz})\text{CH}\cdot\text{C}_6\text{H}_{13}$ , from methyl *n*-heptylketone and benzoyl chloride, b. p.  $210-211^\circ$  (50 m.m.);  $\beta$ -benzoxynonyldecylene ( $\alpha$  or  $\beta$ ),  $\text{CH}_3\text{C}(\text{OBz})\text{CH}(\text{CH}_3)\text{C}_4\text{H}_9$  or  $\text{CH}_3\text{C}(\text{OBz})\text{C}(\text{CH}_3)\text{C}_4\text{H}_9$ , from methyl *sec*-hexylketone and benzoyl chloride, b. p.  $197-200^\circ$  (50 m.m.);  $\beta$ -benzoxynonyldecylene ( $\alpha$  or  $\beta$ ),  $\text{CH}_3\text{C}(\text{OBz})\text{C}_4\text{H}_9$  or  $\text{CH}_3\text{C}(\text{OBz})\text{CH}\cdot\text{C}_3\text{H}_7$ , from methyl *n*-butylketone and benzoyl chloride, b. p.  $170-175^\circ$  (50 m.m.);  $\alpha$ -benzoxynonyldecylene,  $\text{C}_6\text{H}_5\text{C}(\text{OBz})\text{CH}_2\text{C}_6\text{H}_5$ , from acetophenone and benzoyl chloride, b. p.  $227-230^\circ$  (50 m.m.); *1-benzoxycamphene*,—



from camphor and benzoyl chloride, b. p.  $215-220^\circ$  (50 m.m.);  $\alpha$ -benzoxynonyldecylene,  $\text{C}_5\text{H}_{11}\text{CH}(\text{OBz})\text{CH}(\text{OBz})$ , from cinnamaldehyde and benzoyl chloride, b. p.  $195^\circ$  (50 m.m.);  $\beta$ -valeroxynonyldecylene ( $\alpha$  or  $\beta$ ),  $\text{CH}_3\text{C}(\text{O} \cdot \text{CO} \cdot \text{C}_4\text{H}_9)\text{C}_6\text{H}_5$  or  $\text{CH}_3\text{C}(\text{O} \cdot \text{CO} \cdot \text{C}_4\text{H}_9)\text{CH}\cdot\text{C}_6\text{H}_5$ , from methyl *n*-nonylketone and valeryl chloride, b. p.  $185-190^\circ$  (50 m.m.).

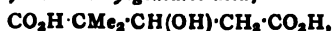
Acetone with benzoyl chloride, and methyl nonylketone with acetyl chloride, did not interact; methyl *n*-propylketone and benzoyl chloride interacted only slightly. All the substances prepared are nearly colourless and odourless oils, and in their chemical properties exactly resemble each other. They all readily absorb bromine, forming dibromo-addition products, and are unstable to potassium permanganate. The possibility of benzoxycamphene having a structure analogous to hydroxycamphene (Forster, *Trans.*, 1902, lxxxi., 264) was considered, but the fact that benzoxycamphene is readily hydrolysed by alcoholic hydroxylamine, yielding *l*-rotatory camphor-oxime, and that its dibromo-addition product readily splits up into  $\alpha$ -bromocamphor and benzoyl bromide, definitely proves the correctness of the structure—



165. "The Synthesis of  $\alpha\alpha$ -Dimethylglutaric Acid, of  $\beta$ -Hydroxy- $\alpha\alpha$ -dimethylglutaric Acid, and of the *cis*- and *trans*-Modifications of  $\alpha\alpha$ -Dimethylglutaconic Acid." By W. H. PERKIN, jun., and Miss A. E. SMITH.

When a mixture of ethyl dimethylmalonate and ethyl acetate is treated with sodium, condensation readily takes place with the formation of ethyl  $\alpha\alpha$ -dimethylacetonedicarboxylate,  $\text{CO}_2\text{Et} \cdot \text{CMe}_2 \cdot \text{CO} \cdot \text{CH}_2 \cdot \text{CO}_2\text{Et}$ , which distils at  $185-190^\circ$  (100 m.m.), and gives, in alcoholic solution with ferric chloride, a red-violet colouration. When this ester is reduced with sodium amalgam and then with hydriodic acid, it yields the following acids:—

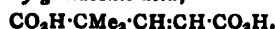
$\beta$ -Hydroxy- $\alpha\alpha$ -dimethylglutaric acid,—



which crystallises from water in prisms, and melts at  $158-160^\circ$ .

$\alpha\alpha$ -Dimethylglutaric acid,  $\text{CO}_2\text{H} \cdot \text{CMe}_2 \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{CO}_2\text{H}$ , which melts at  $90^\circ$ , and is identical with the acid of this constitution obtained from isolauronic acid by oxidation with nitric acid.

*cis*- $\alpha\alpha$ -Dimethylglutaconic acid,—



This acid is readily soluble in water, and melts at  $134^\circ$ ; it combines with bromine, yielding *cis*- $\alpha\alpha$ - $\beta$ -dibromo- $\alpha\alpha$ -dimethylglutaric acid (m. p.  $150^\circ$ ). When hydroxydimethylglutaric acid is distilled, it is, in part, converted into the sparingly soluble *trans*- $\alpha\alpha$ -dimethylglutaconic acid which melts at  $172^\circ$ , and was first obtained (Perkin, *Trans.*, 1902, lxxxi., 253) by the hydrolysis of ethyl  $\alpha$ -bromo- $\alpha\alpha$ -

dimethylglutarate,  $\text{CO}_2\text{Et}\cdot\text{CMe}_2\cdot\text{CH}_2\cdot\text{CHBr}\cdot\text{CO}_2\text{Et}$ , with alcoholic potash. This same acid is obtained almost quantitatively from the above hydroxy-acid by treatment with phosphorus pentachloride, then with alcohol, and finally with alcoholic potash,—



giving  $\text{CO}_2\text{H}\cdot\text{CMe}_2\cdot\text{CH}\cdot\text{CH}\cdot\text{CO}_2\text{H}$ . The *trans*-acid is acted on only with difficulty by bromine with formation of *trans*- $\alpha,\beta$ -dibromo-*aa*-dimethylglutaric acid (m. p. 217°). It appears, therefore, that the *cis*- and *trans*-modifications of *aa*-dimethylglutaconic acid melt respectively at 134° and 172°. The bearing of this on the work of Conrad and of Henrich on this subject was discussed.

166. "A Reaction of some Phenolic Colouring Matters." II. By A. G. PERKIN and C. R. WILSON.

By means of alcoholic potassium acetate, gallacetophenone gives the salts (a)  $\text{C}_8\text{H}_7\text{O}_4\text{K}\cdot\text{H}_2\text{O}$ , and (b)  $\text{C}_{24}\text{H}_{23}\text{O}_{12}\text{K}$ . The former, pale yellow needles, identical with the compound which Nencki and Sieber (*Journ. Prakt. Chem.*, [2], xxiii., 23, 546) prepared with alcoholic potash, is anhydrous at 160°, and gives the salt  $\text{C}_{16}\text{H}_{14}\text{O}_8\text{Ba}$ , yellow needles. Sodium acetate gives the salt  $\text{C}_8\text{H}_7\text{O}_4\text{Na}\cdot\text{H}_2\text{O}$ . With methyl iodide this gives gallacetophenonomethyl ether,  $\text{C}_8\text{H}_7\text{O}_3(\text{OCH}_3)$ , needles, m. p. 132–133°, the diacetyl derivative of which melts at 146–148°; and on methylation gallacetophenonedimethyl ether, m. p. 77–78° (*Trans.*, 1895, lxvii., 997), is formed. It is thus a para- or meta-methoxy-compound. The salt (b), colourless needles, is also formed from gallacetophenone and potassium acetate in aqueous solution, and from this the compound  $\text{C}_{24}\text{H}_{23}\text{O}_{12}\text{Ba}$  can be obtained.

The following salts have been prepared with alcoholic potassium acetate. Ellagic acid gives  $\text{C}_{14}\text{H}_2\text{O}_8\text{K}$  and  $\text{C}_{14}\text{H}_2\text{O}_8\text{K}_2$ , daphnetin,  $\text{C}_9\text{H}_6\text{O}_4\cdot\text{KC}_2\text{H}_3\text{O}_2$ , galangin,  $\text{C}_{15}\text{H}_9\text{O}_5\text{K}\cdot\text{H}_2\text{O}$ , kampheride,  $\text{C}_{16}\text{H}_{11}\text{O}_6\text{K}\cdot\text{H}_2\text{O}$ , dihydroxybenzalcurmaranone ( $\text{OH}:\text{OH}=6:7$ ),  $\text{C}_{15}\text{H}_{10}\text{O}_4\cdot\text{KC}_2\text{H}_3\text{O}_2$ , carminic acid,  $\text{C}_{21}\text{H}_{11}\text{O}_6\text{K}$  and  $\text{C}_{22}\text{H}_{22}\text{O}_{12}\text{Ba}$ , styrogallol,  $\text{C}_{16}\text{H}_7\text{O}_5\text{K}$ , naphthazarine,  $(\text{C}_{10}\text{H}_6\text{O}_4)_2\cdot\text{KC}_2\text{H}_3\text{O}_2?$ , and curcumin,  $\text{C}_{22}\text{H}_{19}\text{O}_6\text{K}$ . By means of alcoholic potash daphnetin gives the salts  $\text{C}_{16}\text{H}_{11}\text{O}_8\text{K}$  and  $\text{C}_9\text{H}_5\text{O}_4\text{K}$ , and carminic acid the salt  $\text{C}_{22}\text{H}_{23}\text{O}_{12}\text{K}$ . From acetyl rhamnetin, the compound  $\text{C}_{16}\text{H}_{11}\text{O}_7\text{K}$  was obtained.

Dimethoxyanhydroglycolgallol,  $\text{C}_{10}\text{H}_{10}\text{O}_4$ , yellow needles, m. p. 122°, gives with protocatechuic aldehyde dimethoxydihydroxybenzalcurmaranone,  $\text{C}_{17}\text{H}_{14}\text{O}_6$ , orange-coloured needles, which with potassium acetate gives the salt  $\text{C}_{17}\text{H}_{13}\text{O}_6\text{K}$ . Pyrogallolmonomethyl ether, incidentally prepared, melts at 102–104°, and not at 95° as given by Benedikt (*Ber.*, 1877, ix., 125).

167. "Note on Mixtures of Constant Boiling-point." By S. YOUNG, D.Sc., F.R.S.

It was noticed by Thorpe (*Trans.*, 1879, xxxv., 544) that when a mixture of equal volumes of carbon tetrachloride and methyl alcohol is distilled, a mixture of minimum boiling-point 55.6–55.9° comes over first. The vapour density of this distillate was found to be 41.82, and the composition was therefore:—

|                      |    |    |    |       |      |
|----------------------|----|----|----|-------|------|
| Carbon tetrachloride | .. | .. | .. | ..    | 78.1 |
| Methyl alcohol       | .. | .. | .. | ..    | 21.9 |
|                      |    |    |    | 100.0 |      |

At Dr. Thorpe's suggestion the author has determined the composition of the mixture of minimum boiling-point by the distillation method, and also from the specific gravity of the mixture after re-distillation.

The results are as follows:—

#### Distillation Method.

|                              | From excess<br>of $\text{CH}_3\text{OH}$ . | From excess<br>of $\text{CCl}_4$ . | From specific<br>gravity. |
|------------------------------|--------------------------------------------|------------------------------------|---------------------------|
| $\text{CCl}_4$ .. ..         | 79.99                                      | 79.35                              | 79.44                     |
| $\text{CH}_3\text{OH}$ .. .. | 20.01                                      | 20.65                              | 20.56                     |
|                              | 100.0                                      | 100.0                              | 100.0                     |

The separation of the mixture of minimum boiling-point from methyl alcohol is very much more difficult than from carbon tetrachloride, and it is in all probability for this reason that the percentage of alcohol in Thorpe's distillate was too high, whilst that calculated from the results of the distillation of a mixture containing excess of alcohol was too low. The most accurate value is probably that deduced from the specific gravity of the re-distilled mixture of constant boiling-point.

Determinations by Thayer (*Journ. Phys. Chem.*, 1898, ii., 382), Ryland (*Amer. Chem. Journ.*, 1899, xxii., 384), and Carveth (*Journ. Phys. Chem.*, 1902, vi., 248) of the boiling-points and composition of mixtures of benzene with ethyl and methyl alcohol were compared with those of Miss Fortey and the author (*Trans.*, 1902, lxxxii., 739).

168. "The Vapour Pressures and Boiling-points of Mixed Liquids." Part II. By S. YOUNG, D.Sc., F.R.S., and Miss E. C. FORTY, B.Sc.

In a previous paper (*Trans.*, 1902, lxxxii., 752) it was shown that, so far as mixtures of chlorobenzene and bromobenzene are concerned, the conclusion of van der Waals is true that if the critical pressures of the two liquids are equal, and if the relation suggested by Galitzine and by Berthelot,  $\alpha_{1,2} = \sqrt{v_{1,2}}$  holds good, the relation between vapour pressure and molecular composition should be represented by a straight line, or the vapour pressures of mixtures may be expressed by the formula—

$$P = \frac{pP_A + (100 - p)P_B}{100},$$

where  $P$ ,  $P_A$ , and  $P_B$  are the vapour pressures of the mixture and of the two components A and B at the same temperature, and  $p$  is the molecular percentage of A.

It does not, however, follow that it is only when the critical pressures are equal that the vapour pressures agree with the formula; indeed, Speyers goes so far as to state that it is always applicable to mixtures of liquids of normal molecular weight.

As the statement of Speyers is certainly too general, it seemed desirable to determine the boiling-points of mixtures of a number of closely related compounds the critical pressures of which are not equal.

Four pairs of such liquids were examined, (1) ethyl acetate and ethyl propionate, (2) toluene and ethyl benzene, (3) *n*-hexane and *n*-octane, (4) benzene and toluene, and, in addition, carbon tetrachloride and benzene.

The investigation resulted in the following conclusions:—

1. The volume and temperature changes on mixing closely related substances are in all the observed cases very small, but it is only with chlorobenzene and bromobenzene that they can certainly be said to be within the limits of experimental error. With the two esters these limits are very closely approached, whilst with benzene and toluene, which are somewhat less closely related, the changes are distinctly larger, and for mixtures of benzene with *n*-hexane and with carbon tetrachloride they are very much larger.

2. The formula—

$$P = \frac{pP_A + (100 - p)P_B}{100}$$

is nearly, if not quite, true for closely related liquids, even when the critical pressures are widely different; it is not true, however, as a rule, for mixtures of liquids of normal molecular weight, such as benzene and *n*-hexane, or for benzene and carbon tetrachloride, which are not closely related.

Of the five pairs of closely related liquids examined, it is only chlorobenzene and bromobenzene which show absolutely no temperature or volume change on mixing, and for mixtures of which the vapour pressures are accurately given by the above formula, and it is only this pair of liquids which have the same critical pressure. The deviations in the other cases are, however, so small that

it remains an open question whether equality of critical pressure is a necessary criterion for absolute agreement with the formula. It appears, at any rate, that equality of critical pressure without closeness of chemical relationship is not a sufficient criterion.

It may be stated definitely that in the case of closely related substances, whatever their critical pressures, the deviations are, as a rule at any rate, exceedingly small.

Lastly, it is shown that there is strong evidence for the existence of mixtures of minimum boiling-point in the case of carbon tetrachloride and benzene, and also of *n*-hexane and benzene, although the boiling-points of the mixtures could hardly be distinguished from those of carbon tetrachloride and *n*-hexane respectively by ordinary thermometric methods.

169. "The Vapour Pressures and Boiling-points of Mixed Liquids." Part III. By S. YOUNG, D.Sc., F.R.S.

If the vapour pressures of mixtures of two liquids, A and B, are accurately expressed by the formula—

$$P = \frac{pP_A + (100 - p)P_B}{100}$$

(where  $P$ ,  $P_A$ , and  $P_B$  are the vapour pressures of the mixture and of the two components respectively at the same temperature, and  $p$  is the molecular percentage of A), and if the difference between  $P_A$  and  $P_B$  is small the relation between the boiling-points of the mixtures at constant pressure and their molecular composition is represented by a nearly straight line, and the temperature may with small error be calculated by means of the formula—

$$t = t_B + \frac{p}{100} (t_A - t_B).$$

The greater the difference between  $P_A$  and  $P_B$ , the greater is the deviation of the boiling-point composition curve from a straight line, and the higher is the molecular percentage of A in that mixture which shows the maximum deviation,  $D$ . This percentage is given with fair accuracy by the formula  $p_D = 50 + 0.18\Delta$  (where  $\Delta$  is the difference between the boiling-points of A and B), and the maximum deviation may be calculated with very slight error from the formula—

$$D_2 = -\frac{0.000158 \Delta^2}{c' \cdot T'} \cdot \frac{c_A}{c_B}$$

where  $c_A$  and  $c_B$  are the values of  $\frac{dt}{dp} \cdot \frac{1}{T}$  for the two components—

$$c' = \frac{(50 + 0.18\Delta)c_A + (50 - 0.18\Delta)c_B}{100}$$

and—

$$T' = \frac{(50 + 0.18\Delta)T_A + (50 - 0.18\Delta)T_B}{100}.$$

The values of  $D_2$ , calculated from the above formula, and of  $D_1$ , deduced from the actual vapour pressures (*Trans.*, 1902, lxxxi., 752) for nineteen pairs of closely related compounds, were shown in a table. The greatest difference between  $D_2$  and  $D_1$  is  $0.16^\circ$ , although the actual value of  $D_1$  is in one case  $-32.8^\circ$ . The boiling-point curves have been determined experimentally in five cases, and the maximum deviation,  $D_3$ , read from the curves; the greatest difference between  $D_3$  and  $D_1$  is  $0.27^\circ$ .

For mixtures of liquids which are not closely related, the formula—

$$P = \frac{pP_A + (100 - p)P_B}{100}$$

is not, as a rule, applicable, but if the ratio  $c_A/c_B$  lies between 0.95 and 1.05 (probably between 0.9 and 1.1),  $D_2$  agrees well with  $D_1$ , and in other cases the difference rarely amounts to one degree. The observed boiling-points of mixtures differ, however, as a rule, from those deduced from the vapour pressures, more especially when the molecules of one or both liquids are associated.

Whether a mixture of constant boiling-point can be

formed or not depends chiefly on the relative values of  $\Delta$  and  $D_3 - D_1$ . If  $\Delta$  is very small, such a mixture may be formed even when the difference between  $D_3$  and  $D_1$  is less than  $1^\circ$ , as in the case of carbon tetrachloride and benzene ( $\Delta = 3.44^\circ$ ,  $D_3 - D_1 = -0.89^\circ$ ). On the other hand, methyl alcohol and water do not form such a mixture, although  $D_3 - D_1 = -4.95^\circ$ ,  $\Delta$  having in this case the high value  $35.11^\circ$ .

170. "Notes on the Condensation Points of the Thorium and Radium Emanations." By E. RUTHERFORD and F. SODDY.

It was shown (*Trans.*, 1902, lxxxi., 342) that the radioactive emanation from thorium compounds passed in unchanged amount through a tube cooled to  $-78^\circ$  with solid carbon dioxide and ether. The acquisition of a liquid air plant has enabled the authors to repeat the experiment at a lower temperature, with the result that they have now succeeded in condensing both the thorium and the radium emanations.

A current of hydrogen (or of air) was passed through the thorium or radium compound, and thence through a copper spiral cooled in liquid air. No trace of emanation escaped in the issuing gas either in the case of radium or of thorium. At the temperature of liquid air the emanations are therefore either condensed or lose their activity.

The radium (or thorium) compound was then removed, and the gas current sent directly through the spiral tube, which was quickly taken out of the liquid air and placed in cotton-wool. Nothing happened for two or three minutes as the temperature of the spiral slowly rose, until suddenly the presence of the emanation was observed in large amount in the escaping gas. The result therefore cannot be explained by supposing that the emanations lose their ionising power at the temperature of liquid air. It is clear that they are condensed and again volatilised. From the sharpness and suddenness of the phenomena there appeared to exist for each emanation a definite temperature of volatilisation. This temperature is very nearly the same in the two cases and considerably above the boiling-point of liquid air.

By measuring the temperature of the copper spiral at the instant of volatilisation, it was found that successive determinations agreed amongst themselves to within a degree. The exact temperatures cannot yet be given, but as a first approximation it may be said that for the radium emanation the volatilisation point lies in the neighbourhood of  $-130^\circ$ , and for the thorium emanation about five degrees lower.

This experiment furnishes an additional proof—if such were needed—that radio-activity is accompanied by the continuous production of special kinds of active matter, which possess distinct and sharply defined chemical and physical properties.

171. "Notes on the Action of Barium Hydroxide on Dimethylvioluric Acid." By Miss M. A. WHITELEY, D.Sc.

When dimethylvioluric acid is decomposed by boiling with barium hydroxide, the chief product is isonitrosomalondimethylamide, which crystallises in well developed prisms belonging to the monosymmetric system ( $a:b:c = 1.53394:1.088680;\beta = 68.43^\circ$ ); it melts at  $157^\circ$ , and yields a yellow potassium salt and a purple ferrous salt, both of which are crystalline. In addition to this compound, six other decomposition products can be isolated, one, melting at  $239-240^\circ$ , has not been identified, the other five are carbon dioxide, oxalic acid (as barium salts), monomethylloxamide, methylamine, and methylamine oxamate. Andreasch (*Monatsh.*, 1895, xvi., 17), who first examined this reaction, obtained only three products, namely, carbon dioxide, methylamine, and a crystalline compound melting at  $228^\circ$ , which he identified as isonitrosomalondimethylamide; this investigation, however, indicates that it was probably an impure specimen of monomethylloxamide.

172. "The Determination of Strychnine and Brucine in *Nux vomica*." By E. DOWDARD.

In order to determine the strychnine, the pure mixed alkaloids are dissolved in 50 c.c. of 2 per cent sulphuric acid, 5 c.c. of nitric acid are added, and the mixture allowed to stand fifteen minutes; the solution is transferred to a separator containing 15 c.c. of ammonia (sp. gr. 0.890) and 10 c.c. of chloroform; the mixture is shaken for five minutes, the solvent separated, and the alkaline liquid again shaken with 10 c.c. of chloroform. The mixed chloroform washings are now shaken with 15 c.c. of dilute ammonia (1 c.c. of above-mentioned strength diluted to 45 c.c.); this is repeated twice, the chloroform evaporated off, and the residue dried at 100° until constant.

To determine the brucine, a standard brucine solution is required, which is made by dissolving 0.16 gm. of anhydrous brucine and 0.16 gm. of strychnine in 2 per cent sulphuric acid, and the solution made up to 100 c.c. Then 0.10 gm. of the pure mixed alkaloids is dissolved in 50 c.c. of 2 per cent sulphuric acid; this solution and 50 c.c. of the standard are placed in two small beakers, 5 c.c. of nitric acid (sp. gr. 1.42) are added simultaneously to the contents of each beaker; the solutions are then transferred to the comparison troughs of a Gallenkamp colorimeter. Five minutes after adding the nitric acid, six readings are taken; from the average of these, the brucine is calculated.

Results were given which proved the accuracy of the determination made by both these methods.

#### PHYSICAL SOCIETY.

Ordinary Meeting, November 28th, 1902.

Prof. S. P. THOMPSON, President, in the Chair.

THE meeting was held, by invitation of Prof. Callendar, in the Physical Laboratory of the Royal College of Science.

Prof. PERRY read a paper on "A Slide-rule for Powers of Numbers."

Soon after the reading of Mr. Lanchester's paper in 1895, "The Radial Cursor; a New Addition to the Slide-Rule," Prof. Perry made slides to assist in computing  $m^n$ , where  $m$  and  $n$  are any numbers. He then came to the conclusion that no great accuracy was obtainable; but on trying the method again he has recently found that it is very convenient and sufficiently accurate for gas- and steam-engine work. These computations can be made with a table of values of  $\log(\log n)$  used in conjunction with an ordinary table of logarithms. In the rule exhibited the D line is replaced by a scale such that the distance from the mark 10 to the mark  $m$  represents  $\log(\log m)$  to the same scale of measurement as that to which the distance from 1 to  $n$  on the C scale represents  $\log n$ . The values of  $m$  range from 2 to 1000, and those of  $n$  from 1 to 10 or from 1 to 0.1 used backwards. The author showed how, with one operation, the rule could be

used to find the value of  $m^n$ ,  $m^{1/n}$ , and the logarithm of any number to any base. If the answer on scale D is less than 2 or greater than 1000, or if the exponent  $n$  is negative, indirect methods involving two operations are necessary. Prof. Perry has replaced the ordinary D line by the log.log scale, because in his opinion this line is the one least used by workers with the slide-rule. The use of the log.log scale was described by Roget in 1814, and the author's object in bringing the matter forward lies in the fact that Dr. Roget's paper seems to be almost unknown, and it is only in these modern days that the computations for which he invented the rule have to be frequently made.

Mr. HARRISON said there could be no doubt that many who had to make calculations would avail themselves of the advantages offered by the addition of the log.log scale to the ordinary slide-rule. He would like to call attention to a further addition by which the general utility of the

log.log scale was increased. It was the introduction of a log. (-log) scale which increased the range of the instrument by making it possible to deal with numbers from a small fraction upwards, with the exception of an unavoidable gap near unity. It also made it possible to directly evaluate a quantity like  $a^{-n}$ . In a slide-rule, if the cursor were misplaced by a small amount, the consequent error in the reading of the log. scale was, for all positions, the same fraction of the number read. On the other hand, in the log.log scale the proportionate error varied along the scale, and was least at the unity end. At the particular place at which the reading is " $e$ " or " $-e$ " the percentage error agreed with that of the comparison log scale. At any other place where the reading was  $e^n$  the error would be  $n$  times this amount. This proportionate accuracy was exactly that which was justified in dealing with experimental numbers liable to a percentage error. It was always possible to increase the degree of accuracy by splitting the numbers involved into suitable factors. Mr. Harrison also gave some account of the preparation of a ten-inch slide-rule accurate to one part in 300, which he hoped it would be possible to sell for one shilling.

Prof. EVERETT said that the slide-rule exhibited by Prof. Perry would be useful for people constantly engaged in raising numbers to strange indices. For occasional use he thought that a table of logarithms was preferable. He pointed out that a complete history of the slide-rule was given in the introduction to the original edition of Hutton's Tables. There were two distinct methods of using logarithmic scales—one due to Gunter and sensibly the same as that employed in Fuller's spiral rule, and the other that used in ordinary slide-rules. With his own form of rule, which he found very convenient, it was possible with care to work correctly to one part in four thousand.

Mr. BOYS remarked that the slide-rule had occupied his attention for many years. The device employed by the author for dealing with powers of numbers was perfectly well known, and the log.log scale was usually referred to as the P line. He admitted that the P line was not so much used as it should be, and hoped that the paper would serve a good purpose in drawing attention to it. He thought, however, that in working with gas- and steam-engines Lanchester's rule was eminently suitable for dealing with cases in which numbers had to be raised to fractional indices. He agreed with Mr. Harrison's remarks upon the accuracy of the exponential scale, but said that they were apt to create a wrong impression. In dealing with an experimental number subject to error the accuracy obtained by the scale was sufficient, but in raising a true number to a certain power the errors introduced were large. He disagreed with Prof. Perry in his assertion that the ordinary D line was less used than the others.

Prof. GREENHILL expressed his interest in the papers and the remarks of Mr. Boys. He thought it would be better to preserve the D line and replace the C line by the log.log scale. He also exhibited the form of Fuller's rule used by the R.H.A.

Mr. APPLEYARD referred to the advantages of the spiral form of slide-rule, and asked if it would be possible to design the sliding part of an ordinary rule on geometrical principles to get rid of the sticking; which frequently occurs.

Mr. COOPER supported Mr. Boys and Prof. Greenhill with reference to the D line, and said he thought it ought not to be abolished.

The CHAIRMAN said an exponential rule was a very useful thing in dealing with some of the calculations connected with alternating currents. A simple method for carrying out a lot of operations for which the slide-rule is often used, would be to commit to memory the multiplication table up to thirty-three times thirty-three.

Prof. H. L. CALLENDAR exhibited a Lecture Experiment for the Determination of the Mechanical Equivalent of Heat.



The experiment was carried out with a modified form of the apparatus exhibited and described by Prof. Callendar at the meeting of the Physical Society held on June 20th, 1902. In the original apparatus conduction from the calorimeter was prevented by ebonite bushes, and the stability of the motion was secured by using a compound belt of silk and leather. In the new pattern the calorimeter is much lighter, and is fastened to driving-cheeks by steel bolts and ivory distance-pieces. The increased conduction loss is compensated by the diminished air loss. Owing to the chemical action of leather on brass, the original belt was removed, and a belt of double and single silk ribbon substituted. A small spring-balance has been introduced to secure stability, and the difference of the weights at the ends of the belt can be read to an accuracy of one part in four thousand. In the actual experiment performed at the meeting, Count Rumford's compensation method was adopted to eliminate the errors arising from loss of heat by radiation and conduction, the calorimeter being half-filled with water at a suitable temperature less than that of the laboratory. The calorimeter was then made to revolve on a horizontal axis by means of an electric motor, and the air-temperature was determined with a platinum thermometer fixed near to the instrument. The experiment was performed by observing the temperature of the water in the calorimeter at a certain instant, and again after every successive 100 revolutions up to 600. The air-temperature was then re-determined, and the mean air-temperature during the experiment calculated. The interval selected for the calculation of  $J$  was from the first reading of the temperature of the calorimeter until the time when the mean temperature of the calorimeter throughout the experiment was the same as the mean temperature of the air. The initial and final air-temperatures were  $19.07$  and  $19.40$ , giving a mean of  $19.24$ . The temperature of the water in the calorimeter at the commencement of the observation was  $16.17$ , after 500 revolutions it was  $21.59$ , and after 600 revolutions  $22.59$ . By interpolation it was found that the mean temperature of the calorimeter throughout the experiment was, after 558 revolutions, the same as the mean air-temperature. The rise of temperature was found to be  $6.00$ , and the value of  $J$  deduced from the experiment was  $4.22$  joules per calorie.

Prof. EVERETT congratulated Prof. Callendar upon the success of the experiment.

The CHAIRMAN expressed his interest in the apparatus and the experiment, and said that a few years ago he would have been surprised at the possibility of determining  $J$  in ten minutes. He thought, however, it was more surprising that it was possible to determine in front of an audience temperatures to within  $\frac{1}{100}$ ° C. This had been rendered possible by the introduction of the platinum thermometer, and he felt that Prof. Callendar had had a large share in bringing that instrument to its present state of perfection.

A paper on "A Portable Capillary Electrometer" was postponed until the next meeting.

The Society then adjourned until December 12th, 1902.

## CORRESPONDENCE.

### "FIXING" NITROGEN FROM THE ATMOSPHERE.

To the Editor of the Chemical News.

SIR,—The atmosphere covering a square mile of the earth's surface would furnish sufficient nitrogen for ten years' supply of sodium nitrate at the rate of 12 million tons of nitrate a year. To put it in another way—Had the fixation of nitrogen from the atmosphere by electrical synthesis and absorption with alkali been commenced some ninety years before the beginning of the Christian

Era and been continued up to the present time, with the same annual output of 12 million tons of sodium nitrate, the effect on the atmosphere would have been so slight as not to show itself by the ordinary methods of gas analysis. In fact, only a millionth of the available nitrogen would have been consumed!

Such a supposed slight deterioration of the atmosphere is, however, founded on an assumption which is more or less questionable, viz., that nitrogen once fixed never finds its way back into the atmosphere.

The same general conclusions apply to oxygen, so that I can scarcely agree with Mr. W. H. Deering in regarding the electrical process of producing nitrates as likely to be a serious draft on the atmosphere.—I am, &c.,

WM. ACKROYD.

Halifax, December 2, 1902.

## THE SUPPOSED DISCOVERY OF ALUMINIUM TWO THOUSAND YEARS AGO.

To the Editor of the Chemical News.

SIR,—Can the quotation in your issue of October 3 (vol. lxxvii., p. 171), regarding the supposed discovery of aluminium two thousand years ago, have reference to the following passage from Pliny's "Natural History," Book XXXVI., Chapter 26? I quote from the classic translation of Philemus Holland.

"Moreover, it is said, That during the reign of Tiberius the Emperor, there was devised a certain temper of glasse, which made it pliable and flexible to wind and turn without breaking: but the artificer who devised this, was put downe, and his work-house, for feare lest vessels made of such glasse should take away the credit from the rich plate of brasse, siluer, and gold, and make them of no price: and verily, this bruit hath run currant a long time (but how true, it is not so certain)."

I have not been able to find any other passage in Pliny which could have given rise to the quotation in the *Globe*. If, however, there be such a passage, the inference need not be drawn that aluminium was known to the ancients. Pliny has, indeed, summed up in his "Natural History" all the knowledge of a scientific character which was to be found in a very complete search of earlier authors, and he supplemented it from his own experience, but unfortunately he did not use any discriminating editorial criticism. The result is a most interesting jumble of science and myth, of fact and fancy, upon which little reliance can be placed.

It is to be hoped that some day a translation of the "Natural History" will be published edited by a competent scientific man.—I am, &c.,

JAS. LEWIS HOWE.

Washington and Lee University,  
Lexington, Virginia, November 19, 1902.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxxv., No. 20, November 17, 1902.

Impurities of Compressed Oxygen and their Effect on Combustions in the Calorimetric Bomb.—M. Berthelot.—When oxygen is used in the calorimetric bomb for determining heats of combustion, it is essential that the gas should be free from all combustible matter. Water vapour and carbon dioxide (the latter in small traces) do not interfere with calorimetric measurements. In many cases it is advantageous to saturate the oxygen with water

vapour before the combustion, as it renders negligible the calorific effect due to reduction to vapour of the water produced by the combustion itself. Traces of nitrogen present in the oxygen cause nitric acid to be produced, and an error occurs in the result in consequence. The presence of hydrogen or combustible gases has also to be allowed for. It is necessary to make all junctions with metallic copper without using any organic materials. In one experiment it was found that the quantity of oxygen introduced into the bomb contained practically no carbon dioxide, but 0.0005 grm. of free hydrogen. This weight is susceptible of developing 17.2 cal. of heat during its combustion.

**Atmospheric Hydrogen.**—Anatole Leduc.—The author's experiments lead him to the conclusion that former researches on the proportion of hydrogen in the atmosphere cannot be correct.

**The Oxalomolybdates.**—M. Bailhache.—The author prepares complex oxalates from sulphate of molybdenum,  $\text{Mo}_2\text{O}_5 \cdot 2\text{SO}_3$ , and examines their reactions. If these react on sodium acetate in a current of hydrogen, a precipitate is formed. This, when washed with alcohol and dried, gives proportions of metal and oxygen which are not constant. This fact can be explained by the supposition that the salt is a variable mixture of hydrated molybdenum dioxide and another hydrate,  $\text{Mo}_2\text{O}_5 \cdot 3\text{H}_2\text{O}$ . The reactions obtained by an aqueous solution of molybdenum sulphate confirm this theory. When ammonium chloride is added and the solution saturated with hydrochloric acid gas, green crystals of double chloride of molybdenyl and ammonium are deposited. Oxalomolybdate of potassium also gives this characteristic reaction when dissolved with ammonium chloride in strong hydrochloric acid—



## MISCELLANEOUS.

**Royal Institution.**—The following are the Lecture Arrangements at the Royal Institution, before Easter:—

Professor H. S. Hele-Shaw, Six Lectures (adapted to young people) on "Locomotion—on the Earth; through the Water; in the Air"; experimentally illustrated.

Professor Allan Macfadyen, Fullerian Professor of Physiology, R.I., Six Lectures on "The Physiology of Digestion."

Sir William Abney, Three Lectures on "Recent Advances in Photographic Science."

Sir Robert Ball, Three Lectures on "Great Problems in Astronomy."

Mr. A. J. Evans, Three Lectures on "Pre-Phœnician Writing in Crete, and its bearings on the History of the Alphabet."

Sir Clements Markham, Three Lectures on "Arctic and Antarctic Exploration."

Mr. G. R. M. Murray, Three Lectures on "The Flora of the Open Ocean."

Mr. C. H. Firth, Three Lectures on "Society during the Commonwealth and Protectorate."

Sir Frederick Bridge, Three Lectures on "The Bicentenary of Samuel Pepys—His Musical Contemporaries, Criticisms, and Compositions" (with Musical Illustrations).

Mr. A. B. Walkley, Three Lectures on "Dramatic Criticism."

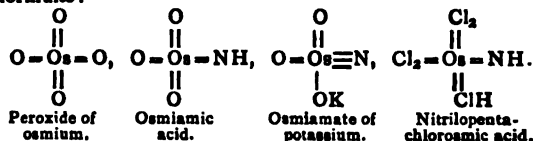
Six Lectures by The Right Hon. Lord Rayleigh.

The Lectures on Tuesdays and Thursdays during the Season 1903 will be delivered at Five o'clock instead of at Three, as in previous years. The Christmas Course of Juvenile Lectures and the Saturday Lectures will continue to be delivered at three o'clock.

The Friday Evening Meetings will begin on January 12th, when a Discourse will be delivered by Professor

Dewar on "Low Temperature Investigations"; succeeding Discourses will probably be given by Dr. Tempest Anderson, Professor W. E. Dalby, The Right Hon. Sir Herbert Maxwell, Bart., M.P., Professor S. Delépine, Principal E. H. Griffiths, Dr. A. Liebmans, Professor J. G. McKendrick, Professor Karl Pearson, Professor E. A. Schäfer, Professor W. A. Herdman, The Right Hon. Lord Rayleigh, and other gentlemen.

**The Nitrilopentachlorosmiate, and on the Constitution of Osmiamic Acid.**—A. Werner and K. Dinklage.—We treat 1 grm. of finely powdered osmium acid, cooling and stirring the whole time; chlorine is given off. After half-an-hour crusts of red crystals are deposited, which are purified by draining, re-dissolving in a little water, and re-precipitating with hydrochloric acid. In this manner we obtain a reddish brown crystalline powder, soluble in water, with a cherry-red colour, but decomposed, giving a brown precipitate, after prolonged contact with water. It is insoluble in the organic solvents; on boiling with potash there is no disengagement of ammonia. The formula of this body is  $\text{OsNCl}_5\text{K}$ , and the authors call it *nitrilopentachlorosmiate of potassium*; the mother-liquors contain the corresponding free acid,  $\text{OsNCl}_5\text{H}$ , and the reaction can be explained in the following manner by admitting that a monopotassic salt is formed first:  $\text{OsNO}_3\text{K} + 7\text{HCl} = \text{OsNCl}_5\text{KH} + \text{Cl}_2 + 3\text{H}_2\text{O}$ . The mother-liquors, treated with chloride of ammonium, precipitate a violet microcrystalline powder, which is the corresponding ammoniac salt,  $\text{OsNCl}_5\text{H} \cdot \text{NH}_4$ , similar to the potassic salt. In the same way we can get the salts of caesium and rubidium; they are still less soluble, especially the latter. The authors think that the osmium is octavalent, and propose the following constitutional formulæ:—



—*Berichte*, vol. xxxiv., p. 2698.

## MEETINGS FOR THE WEEK.

**MONDAY, 15th.**—Society of Arts, 8. (Cantor Lectures). "The Future of Coal-gas and Allied Illuminants," by Prof. Vivian B. Lewes.

**WEDNESDAY, 17th.**—Chemical, 5.30. "A Reagent for the Identification of Carbamide and of certain other Nitrogen Compounds," by H. J. H. Fenton. "The Rate of Decomposition of Diazo-compounds—Part II., Diazo-compounds of the Naphthalene Series," by J. C. Cain and F. Nicoll. "The State of Carbon Dioxide in Aqueous Solution" and "Qualitative Separation of Arsenic, Antimony, and Tin," by J. Walker. "The Hydrates and Solubility of Barium Acetate," by J. Walker and W. A. Fyfe. "The  $\gamma$ -Dimethylglutaric Acids, and the Separation of *cis*- and *trans*-forms of Substituted Glutaric Acid," by J. F. Thorpe and W. J. Young.

Microscopical, 8. "The Genus *Diaschisma*," by F. R. Dixon-Nuttall and Rev. R. Freeman. Demonstration on a New Arrangement for taking Photomicrographs in Colours, by E. R. Turner.

Society of Arts, 8. "The South Russian Iron Industry," by Archibald P. Head, M.Inst.C.E.

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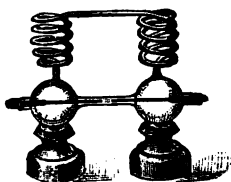
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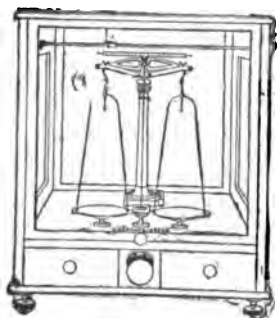
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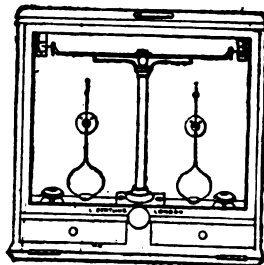


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# THE CHEMICAL NEWS.

VOL. LXXXVI., No. 2247.

## SOME REMARKS ON THE CONNECTION BETWEEN METALS AND NON-METALS.

By GREGORY MARTIN, B.Sc.(Lond.).

A GENERAL study of the Periodic Table shows that in any one group an increase of atomic weight almost invariably causes metallic properties to appear. This is well seen in the case of the group elements, C, Si, Ge, Sn, Pb, of Group IV., and N, P, As, Sb, Bi, of Group V.

In each case it seems that there is a gradual transition, with increase of atomic weight, of the non-metals into metals. The same relationship holds in almost all the other groups.

Now, it is a very remarkable fact that, in the case of the elements of the odd series at any rate, when an increase of atomic weight brings out metallic properties, it produces also at the same time a similar effect on the chemical behaviour of the elements as an increase of temperature.

To illustrate this, let us take the case of the elements C, Si, Ge, Sn, and Pb.

Carbon and silicon at ordinary temperatures are very stable in a tetravalent condition, and extremely unstable in a divalent state. But as the atomic weight increases, the divalent condition becomes more and more stable, and the tetravalent condition more and more unstable, until finally, in the case of lead, the divalent condition is far more stable than the tetravalent condition, as appears at once manifest on contrasting  $PbCl_4$  with  $PbCl_2$ , and  $PbO_2$  with  $PbO$ .

Now, this is precisely the same effect as an increase of temperature alone would bring about. Almost invariably the higher the temperature the more and more do elements tend to pass from a higher to a lower grade of valency.

Iron, for example, at ordinary temperatures is very stable in the  $Fe^{III}$  state, and unstable in the  $Fe^{II}$  state, as at once appears when we contrast  $FeCl_3$  with  $FeCl_2$ . But at a very high temperature the  $Fe^{III}$  state becomes unstable, and the  $Fe^{II}$  state stable, the  $FeCl_3$  passing spontaneously into  $FeCl_2$ . This is the case with most other elements also; in fact, it is a general phenomenon.

On reflecting on these matters, it occurred to me early in this year that if an increase of atomic weight acts in the same way as an increase of temperature, would not, conversely, an increase of temperature act in the same way as an increase of atomic weight?

So that if an increase of atomic weight causes non-metals to pass gradually into metals, would not also an increase of temperature produce the same effect?

In other words, would substances that are non-metals at ordinary temperatures assume metallic properties at a very high temperature?

If this be so, a non-metal is nothing more or less than a substance viewed at a temperature *too low* for it to assume metallic properties; and, conversely, a metal is a substance viewed at a temperature *too high* for it to assume non-metallic properties.

So that the metallic and non-metallic conditions are simply *phases*, which all kinds of matter pass through as the temperature increases from zero upwards. If, therefore, we could only cool down a metal, such as copper, to some exceedingly low temperature it would become a non-metal, just as glass is at ordinary temperatures, and refuse to conduct electricity; while, conversely, if we took the non-metallic carbon and heated it up to an excessively high temperature, it would become a metal and conduct electricity.

Of course, this is a mere speculation. But the idea is a

very fascinating one indeed, and, if true, opens out a whole vista of possibilities hitherto scarcely dreamt of.

Moreover, it is contradicted by no known fact. On the contrary, there exist a great and growing number of facts which appear favourable to it. For instance, we all know how enormously the electric conductivity of non-metals increases as the temperature rises. It is sufficient to recall here the enormous increase in the conductivity of the rare earths at a temperature above  $800^\circ$ , a fact which Professor Nernst has utilised in his celebrated lamp. Carbon also increases enormously in conductivity. Professor T. Gray (*Proc. Roy. Soc.*, January 12, 1882) showed that an increase of only  $300^\circ$  C. caused the conductivity of the best insulating flint glass to increase nearly eight-thousand-fold!

Conversely, Callendar and Dewar have established the fact that the electrical resistance of metals does not continue to decrease as the temperature falls, but reaches a small and nearly constant value (Callendar, *Phil. Mag.*, 1899, xlvii., 217), and Lord Kelvin believes (*Phil. Mag.*, March, 1902) that a metal below  $1^\circ$  absolute is a perfect insulator of electricity, at  $2^\circ$  it shows noticeable conductivity, and at  $6^\circ$  it possesses high conductivity; in other words, he thinks that a metal at these low temperatures behaves precisely as a non-metal behaves at ordinary temperatures.

It may of course be objected that whereas the resistance of metals at ordinary temperatures steadily increases with rise of temperature, the resistance of non-metals continually diminishes, so that both kinds of matter act in this respect in a directly opposite manner.

But it must be remembered that our experiments in the case of non-metals have as yet been made over very limited temperature ranges. The molecules of metals, for example, at ordinary temperatures consist of only single atoms; whereas those of non-metals consist almost invariably of several atoms bunched together. Before we can expect non-metals to behave like metals, we should have to heat them so highly that they too are reduced to the same molecular condition as metals at ordinary temperatures; and this only occurs at very high temperatures indeed, as Victor Meyer proved in the case of the halogens.

This idea of the mutual convertibility of metals and non-metals might appear at first sight somewhat startling. But this is due in great measure to the fact that we have been educated to regard metals and non-metals as two distinct and discontinuous kinds of matter. Yet this is not the case. Metals and non-metals are not separated by any arbitrary line of demarcation, but shade gradually and imperceptibly into each other. And this seems to proclaim the unity of both kinds of matter. Why, therefore, should they not be mutually inter-convertible?

It must be remembered the history of science is full of the gradual fall of all arbitrary lines of demarcation. For example, before the time of James Thomson the melting-point of ice was thought to be one of the eternal and unchangeable constants of nature. Yet we all know now that, like most other "constants," it is in truth a variable, depending upon the external conditions of temperature and pressure.

So also at the present time, the metallic or non-metallic nature is thought to be an unchangeable characteristic of an element. So that if an element *happens* at ordinary temperatures and pressures to exhibit metallic or non-metallic properties, as the case may be, it will continue to exhibit them under *all* other conditions of temperature and pressure.

Yet such a view is extremely improbable, and it gains nothing in probability when we reflect on the profound and indeed startling way the properties of every element changes with the temperature and pressure. For instance, carbon at ordinary temperatures is a perfectly inactive element. It refuses to combine with any other element, nor will it dissolve even in the strongest acids.

It is, in fact, an inactive body like nitrogen. Yet it



completely changes its nature at a high temperature. At a red or white heat it becomes one of the most chemically active elements known, displacing metals from their combination with oxygen, and freely combining with H, S, and O.

While at the temperature of the electric arc, it attacks indiscriminately the metals, dissolving freely in them to form the somewhat indefinite carbides, much as metals dissolve in each other at low temperatures to form alloys.

Other elements, too, change their nature in a way quite as remarkable; sodium, for example, being quite chemically inactive at  $-80^{\circ}\text{C}$ .

With these astonishing modifications in the properties of elements in mind, he would indeed be bold who would venture to deny that it is possible for a non-metal to change into a metal at a high temperature, or a metal into a non-metal at a low temperature.

A theoretical explanation follows directly from the theory of electrons. We must suppose that non-metals possess a greater specific attraction for electrons than metals, both being viewed at a common temperature.

So that in the case of substances which are in a non-metallic state, the temperature at which they are viewed is not sufficient to detach the electrons from their atoms, and cause them to wander freely in the inter-molecular interstices. Whereas, in the case of substances which are in a metallic state, the temperature is high enough.

In the first case (non-metals), an electric current is caused by the movement of electrons and the atoms to which they are attached (electrolytic conduction). Whereas, in the second case, the electrons move independently of the atoms ("metallic" conduction).

An increase of electric tension acts in much the same way as an increase of temperature in detaching electrons from the surfaces of atoms. So that during the passage of a disruptive discharge, a non-metal, such as glass, is converted into a metallic state. An increase of the electric tension, therefore, lowers the temperature at which non-metals pass into metals. The temperature of conversion is lowered to the ordinary temperatures at the point when the dielectric breaks down under the electric strain.

University of Kiel, Nov. 28, 1902.

### THE ESTIMATION OF SULPHUROUS ACID BY MEANS OF A TITRATED SOLUTION OF IODINE.

By A. BERG.

In the year 1887, in a paper on the examination of the estimation of sulphurous acid by Bunsen's method, M. Volhard made a series of careful observations on the reactions produced when solutions of hydriodic acid and sulphurous acid are added together; in such cases a yellow colouration is produced. According to M. Volhard (*Liebig's Annalen*, vol. cxliii., p. 93) this colouration indicates the formation of hydrosulphurous acid. I am unable to agree with this opinion, and from the facts which I am about to describe I am more inclined to adopt the views of M. Péchard. This chemist (*Comptes Rendus*, vol. cxxx., p. 1188), by allowing gaseous sulphurous anhydride to act on dry iodide of potassium, obtained a yellow compound, of which he measured the tension of dissociation, and of which the formula is  $\text{KISO}_2$ . M. Péchard is of the opinion that the same compound is produced still when we operate in the presence of water.

For the purpose of proving the presence of hydrosulphurous acid in the yellow solutions, M. Volhard relies on:—(1) The colour; (2) that, according to him, they have reducing powers more strongly marked than in the corresponding solutions of sulphurous acid; they de-

colorise solutions of indigo instantaneously (*Bull. Soc. Chim.*, [3], vol. xxiii., p. 673).

It is easy to see that the solution of hydrosulphurous acid, such as is obtained by shaking up a solution of sulphurous acid with zinc, is of a reddish-yellow, and not of a greenish-yellow colour like the mixtures of solutions of sulphurous acid and hydriodic acid or iodides.

As for the reducing properties, I have observed that there is a great difference between the solution of hydrosulphurous acid, which decolorises instantly a fairly dark coloured solution of indigo with two or three drops, and the solutions of sulphurous acid treated with hydriodic acid or iodide, which only decolorises it partially, and not sensibly more than does sulphurous acid alone.

To these differences between the two kinds of solutions we can now add one still more important, and that is their action on iodomercurate of potassium. Hydrosulphurous acid reduces it immediately when only a small amount is used, and gives a grey precipitate of mercury, while the other solutions are absolutely without action, no matter in what proportion they are used.

The above facts, if they do not enable us to affirm that the yellow colouration is due to the formation of a compound analogous to that obtained by M. Péchard, still entitles us to doubt the presence of hydrosulphurous acid in this aqueous mixture.

There is another point in M. Volhard's work upon which I cannot agree with him; we all know the method of estimating sulphurous acid by means of a titrated solution of iodine devised by Bunsen. We are also aware that this writer only claims accuracy for his method when carried out with very dilute solutions of sulphurous acid (0.03 to 0.04 per cent). With more concentrated solutions the method gives lower figures than we should expect to find theoretically, a fact that Bunsen attributes to the reaction brought about between the sulphuric and hydriodic acids first formed. Several writers have examined this question; most of them have confirmed the irregularities observed by Bunsen without admitting the explanation he gave of the phenomenon; others look upon the method as exact; all agree, however, on this point, namely, that theoretical results are obtained if the sulphurous acid is dropped into the iodine solution instead of the other way about.

After having verified the above irregularities, which, according to his researches, might be as much as a loss of one quarter of the sulphurous acid present in solutions at 2 per cent, Volhard explains the fact by the decomposition, during the titration, of a part of this body into sulphur and sulphuric acid under the influence of the hydriodic acid which is formed. In fact, he has shown that this decomposition would take place according to the following formula:  $-\text{3SO}_2 + 2\text{H}_2\text{O} = \text{S} + 2\text{SO}_4\text{H}_2$ .

Now, in a research on the same subject (*Bull. Soc. Chim.*, [3], vol. xxiii., p. 499) I became thoroughly convinced of the slowness of this decomposition, since, when we mix solutions of sulphurous acid and hydriodic acid under more favourable conditions than those realised by titration, not less than four or five days elapse before the solution becomes cloudy on account of the precipitation of the first portions of the sulphur. From this it appears to be very improbable that this decomposition could interfere with and falsify the estimation.

Further, will not the two following facts explain the anomalies in a far simpler manner?

(1) Sulphurous acid being oxidisable on contact with the air, this oxidation may take place during the titration; (2) solutions of sulphurous acid, even when dilute, give off a strong odour of this body, which indicates its diffusion into the atmosphere, and consequently its partial disappearance from the solution.

It was on this account that I undertook the examination of Bunsen's method, guarding against these causes of error, and I proceeded in the following manner:—

A Woolf bottle of 500 c.c. capacity was fitted by means of rubber stoppers in the side necks with two burettes

with taps, containing respectively the sulphuric acid to be examined and the titrated solution of iodine. Through the centre neck two tubes were fitted, by which means a current of carbonic acid could be passed through the flask. These tubes were bent three times at right angles outside the flask, their terminal lengths being horizontal, and each one was fitted with a piece of indiarubber tube and a pinchcock; this portion was plunged into boiled water.

After having driven the air out of the apparatus by means of the carbonic acid, the tube through which the gas arrives is closed by screwing up the pinchcock; aspiration is effected by means of the second tube so as to diminish the pressure in the flask sufficiently to allow the liquids in the burettes to run into the flask; finally, the second pinchcock is closed.

We begin by allowing a known volume of the sulphurous solution to run in, then the iodine liquor until there is a little free iodine which does not disappear on prolonged agitation. The whole of the sulphurous acid, both that in solution and that diffused in the atmosphere of the apparatus, has disappeared by this time.

The pinch-cocks are unscrewed, and this allows the boiled water to enter the tubes and drive out the small amount of sulphurous acid which might possibly be there. The excess of iodine is then estimated by means of hypophosphite.

The operation is repeated, but this time a volume of iodine solution is run in first, more than sufficient to oxidise the sulphurous acid which is introduced afterwards. Under these conditions we know that the estimation is correct.

I have used this method with solutions of sulphurous acid of various strengths, and using iodine solutions of different values, according to circumstances, from one-fifth to  $\frac{1}{125}$ th of an atom of this metalloid per litre.

In the following table the volumes of the titrated solution used are expressed in iodine solution at one-tenth of an atom, or 12.7 grms. per litre, and correspond with 10 c.c. of sulphurous solution:—

| Titration, adding the SO <sub>2</sub> to the I. |                                 | Titration, adding the I to the SO <sub>2</sub> . |                            |                                 |
|-------------------------------------------------|---------------------------------|--------------------------------------------------|----------------------------|---------------------------------|
| SO <sub>2</sub> per cent taken.                 | Volume of iodine solution. C.c. | Time of titration. Minutes.                      | Volume of I solution. C.c. | SO <sub>2</sub> per cent found. |
| 0.105                                           | 3.27                            | 10                                               | 3.35                       | 0.107                           |
| 0.518                                           | 16.2                            | 10                                               | 16.0                       | 0.512                           |
| 0.998                                           | 31.2                            | 10                                               | 31.3                       | 1.00                            |
| 1.93                                            | 60.2                            | 10                                               | 60.4                       | 1.99                            |
|                                                 |                                 | 45                                               | 60.6                       | 2.06                            |
|                                                 |                                 | 10                                               | 176.0                      | 5.74                            |
| 5.62                                            | 175.6                           | 45                                               | 177.2                      | 6.13                            |

By an examination of the above figures we see that the concordance of the results given by the two methods of working is very satisfactory for concentrations below 1 per cent.

For higher concentrations the agreement is not so close, but curiously enough it is in the opposite direction to what would be expected, more sulphurous acid was found than was actually present in the solution. I have ascertained without any doubt that this is due to the small amount of sulphurous acid which remains between the plug of the glass tap and the tip of the tube, giving off sulphurous acid fumes during the whole time of the titration; this is naturally added to that in the solution used. Again, it may be remarked that the difference is the greater as the time of the titration is longer.

For the purpose of removing this cause of error, I modified the method of working by substituting a sealed tube for the burette containing the sulphurous solution; this tube was drawn out fine at the extremities, and it contained a known weight of the solution under examination. The upper end of the tube was capped with a piece of indiarubber carrying a small pinchcock; owing to the elasticity of the indiarubber stopper, the lower point can

be broken off against the inside of the neck of the flask, after all the air has been driven out of the latter by a current of carbonic acid gas, and a partial vacuum made; the upper point in the indiarubber tube is then broken, and by connecting this latter with the carbonic acid generator and unscrewing the pinchcock the liquid is run into the flask. The operation is then finished as above, taking care at the end to pass some of the boiled water through the drawn-out tube, so as not to lose any trace of sulphurous acid.

In this manner the following results were obtained:—

| Pouring the SO <sub>2</sub> into the I. |                            | Pouring the I into the SO <sub>2</sub> . |                            |                                 |
|-----------------------------------------|----------------------------|------------------------------------------|----------------------------|---------------------------------|
| SO <sub>2</sub> per cent taken.         | Volume of I solution. C.c. | Time of titration. Minutes.              | Volume of I solution. C.c. | SO <sub>2</sub> per cent found. |
| 5.35                                    | 167.4                      | 45                                       | 167.0                      | 5.34                            |
| 6.82                                    | 213.2                      | 45                                       | 213.6                      | 6.83                            |

Thus we see that a very close concordance was obtained between the figure obtained by the two estimations, no matter what the concentration of the sulphurous acid is (up to about 7 per cent), or the time taken up by the titration (up to forty-five minutes).

Thus Bunsen's method gives exact results, if care be taken to exclude the air and to prevent all loss of sulphurous acid by diffusion. There remains to decide to what extent each of these causes of error is responsible.

1. *Oxidation*.—To examine the influence of oxidation, I repeated the same titrations as before, but left the flask full of air. As was to be expected, the figures obtained in this manner varied with the length of the operation and the more or less prolonged shaking up of the solutions. I will only give a few of the results, to show approximately the extent of the discordances observed:—

| Pouring the SO <sub>2</sub> into the I. |                            | Pouring the I into the SO <sub>2</sub> . |                                           |                                    |  |
|-----------------------------------------|----------------------------|------------------------------------------|-------------------------------------------|------------------------------------|--|
| SO <sub>2</sub> per cent taken.         | Volume of I solution. C.c. | Volume of I solution. C.c.               | Loss of SO <sub>2</sub> per 10 c.c. Grms. | Loss per cent of SO <sub>2</sub> . |  |
| 5.62                                    | 175.6                      | 170.2                                    | 0.0173                                    | 3.0                                |  |
| 1.98                                    | 60.2                       | 58.8                                     | 0.0044                                    | 2.3                                |  |
| 0.998                                   | 31.2                       | 30.8                                     | 0.0013                                    | 1.3                                |  |

These figures show that the oxidation, while not being negligible, is not the principal cause of the irregularities observed by different writers. I expected to find that this oxidation would be much more important, for if it is the fact that sulphurous acid in solution oxidises slowly in contact with air, I have shown that this oxidation becomes much more rapid in the presence of iodides or of hydriodic acid. To make quite certain of this, it suffices to shake up, in two identical flasks full of air, on the one hand, 10 c.c. of a solution of sulphurous acid, and, on the other hand, the same quantity of the solution treated with 1 grm. of iodide of potassium. After ten minutes, if a salt of barium is poured into each of the flasks, a faint cloudiness is produced in the former, while an abundant precipitate of sulphate of baryta is formed in the latter.

If the above operation is carried out in a graduated test-glass containing a known volume of air, we observe that the absorption of oxygen is about thirteen times as great in the presence of the iodide as in its absence.

This is an interesting phenomenon which I propose to examine further, later on.

2. *Diffusion*.—This is the principal cause of the errors of estimation. To estimate it I placed 10 c.c. of a sulphurous solution, of which the strength was known, in a wide-mouthed conical flask, and for one minute I gave the solution the gyratory movement analogous to that given during titration. After having blown gently into the flask to renew the atmosphere, I rapidly poured the contents as well as the washings with boiled water into an excess of iodine solution. In this manner I obtained the following figures, which again are only approximate:—

| Pouring the SO <sub>2</sub> into the I. |                            | Pouring the SO <sub>2</sub> into the I, after agitation. |                                     |                                  |
|-----------------------------------------|----------------------------|----------------------------------------------------------|-------------------------------------|----------------------------------|
| SO <sub>2</sub> per cent taken.         | Volume of I solution. C.c. | Volume of I solution. C.c.                               | Loss of SO <sub>2</sub> per 10 c.c. | Loss per cent of SO <sub>2</sub> |
| 5.62                                    | 175.6                      | 97.0                                                     | 0.251                               | 44.7                             |
| 1.93                                    | 60.2                       | 40.6                                                     | 0.0627                              | 32.5                             |
| 0.998                                   | 31.2                       | 21.8                                                     | 0.0299                              | 30.0                             |
| 0.518                                   | 16.2                       | 12.4                                                     | 0.0125                              | 24.1                             |
| 0.1056                                  | 3.30                       | 2.70                                                     | 0.0019                              | 18.1                             |
| 0.0423                                  | 1.27                       | 1.16                                                     | 0.0006                              | 13.8                             |

We can see of what great importance is the loss of the sulphurous acid under the above conditions, since it attains almost 50 per cent with solutions at about 6 per cent strength, and is still 14 per cent with very dilute solutions of 0.04 per cent.

Contrary to the hypothesis of M. Volhard, the decomposition of the sulphurous acid into sulphur and sulphuric acid does not interfere in any way with the estimation. The same may be said of the reaction of sulphuric acid on hydriodic acid mentioned by Bunsen. The only causes of error in the estimations are oxidation and diffusion of the sulphurous acid on contact with the air, causes which can be prevented by pouring the sulphurous acid into the iodine solution.—*Bull. Soc. Chim.*, Series 3, vol. xxvii., No. 22.

#### COMMERCIAL VALUE OF SCIENTIFIC KNOWLEDGE.

##### RESEARCH CHEMISTS TO BE APPOINTED BY THE GAS LIGHT AND COKE COMPANY.

In his Presidential Address to the Royal Society, delivered at its annual meeting on December 1st, Sir William Huggins joined the chorus of eminent persons who, during the past year or two, have been endeavouring to impress upon the business men in this country the absolute necessity of a more systematic application of scientific methods, in theory and in practice, to our manufactures and industries. To the plea for increased attention to science in all departments of business, we have from time to time lent whole-hearted support—always giving prominence to the opinion, which we hold very strongly, that in regard to educational reform the impetus necessary in this slow-moving country to bring about any really effective improvement must come from business men, captains of industry. If only employers will (in the language of the advertisements) ask for well-educated employees and see that they get them, the steps necessary for the production of a larger supply of intelligently and usefully educated men will very soon be taken. Then, and only then, will what Sir William Huggins rightly described as "the present inappreciative attitude of our public men, and of the influential classes of society generally, towards scientific knowledge and methods of thought," be radically changed.

Holding this view, we are pleased to give publicity to what we understand is the fact, that the Directors of the Gas Light and Coke Company have recently decided to appoint one or more Research Chemists to carry out investigations and experiments at their extensive chemical works at Beckton, with a view to the more profitable utilisation of the products of coal-tar distillation and the other processes now carried out at these works. This is a very welcome move in the right direction. It has long been a reproach to this country that our German neighbours buy our crude tar products and make enormous profits out of the dyes and other articles of commerce manufactured from these products; and it was only in the October of last year that we devoted several columns of the *Journal of Gas Lighting* to an exposition—first, of the enormous progress of the chemical industries in Germany during the past quarter of a century, and second

of the magnificent system of education which has given Germany the supply of trained chemists which has enabled her to achieve that progress. For the five years 1895-9, three of the largest firms in the dye-manufacturing industry in Germany paid not less than 24 per cent, not less than 26 per cent, and 18 per cent per annum dividends respectively, on capitals amounting in the aggregate to nearly £2,500,000. The total annual production of the German chemical works is now about £50,000,000; and imports are decreasing and exports increasing. Why should this enormous volume of trade and so substantial a percentage of profit from the manufactured articles, for which this country supplies much of the raw material, be allowed to rest quietly in the hands of our German trade competitors? That, apparently, is the question which the Horseferry Road authorities are seeking to answer; and we heartily wish them success in their enterprise, which, as an example to other leaders of industry, is of more value than any amount of talking or writing.

If, however, we are to wrest trade from our rivals, or prevent them from seizing ours, we must learn the lesson from Germany that this is only to be done by paying great attention, and devoting large sums of money, to the provision of facilities for obtaining thorough scientific education. Germany has risen to the first rank in chemical industry by means of the men who have been given thorough instruction, both theoretical and practical, at her Universities and Technical High Schools; and we can only fight science with science. We may repeat the question with which we concluded the articles to which reference has been made: Has this country, has the gas industry, nothing to learn from a consideration of the past successes, the present position, and the future prospects of chemical research in Germany?—*Journal of Gas Lighting*.

**American Philosophical Society for Promoting Useful Knowledge.**—The success of the General Meeting of the American Philosophical Society, held at Philadelphia last April, has established satisfactorily the claim that the interests of useful knowledge in the United States may be greatly promoted by holding an annual General Meeting of the Society. Such a meeting, not only from the information derived from the papers presented, but also from their discussion, has proved attractive to its members from all parts of the country and has broadened the field of usefulness of this, the oldest scientific society in America. At the concluding session of the General Meeting held last April it was resolved that a second General Meeting be held in April, 1903. In accordance with this resolution the said General Meeting of the Society will take place on Thursday and Friday, April 2 and 3, 1903, and a Committee has been appointed to make the necessary arrangements. Members desiring to present papers, either for themselves or others, are requested to send to the Secretaries at as early a date as practicable and not later than March 1, 1903, the titles of these papers, accompanied by a brief abstract, so that they may be duly announced on the programme which will be issued immediately thereafter, and which will give in detail the arrangements for the meeting. Papers in any department of science come within the scope of the Society, which, as its name indicates, embraces the whole field of useful knowledge. The Publication Committee, under the rules of the Society, will arrange for the immediate publication of the papers presented. The Society by means of its publications, which present a series covering 140 years and include *Transactions* in quarto and *Proceedings* in octavo, with its large exchange list embracing, practically, the scientific societies of the world, and with its exceptional facilities for immediate issue, offers unexcelled avenues for prompt publication and wide circulation of the papers read before it.

CONTRIBUTIONS TO OUR KNOWLEDGE OF  
YTTERBIUM.\*

By ASTRID CLEVE.  
(Continued from p. 287).

III. COMPOUNDS OF YTTERBIUM (continued).

Ytterbium Meta-tungstate,  $\text{Yb}_2\text{O}_3 \cdot 12\text{WO}_3 + 35\text{H}_2\text{O}$ .

This was obtained from ytterbium sulphate and barium meta-tungstate. The salt crystallises from concentrated solutions in transparent prisms bounded by rectangular surfaces. It is very readily soluble, but is not deliquescent, and, on the whole, is stable in the air.

Analysis of the Pressed Salt.

- 2.2000 grms. of the substance lost 0.1712 gm. over sulphuric acid, and 0.0660 gm. more at  $115^\circ$ . 0.4617 gm. of the residue gave after ignition 0.4390 gm. of the anhydrous salt.
- 0.8571 gm. of the substance on ignition gave up 0.1396 gm. of water, and yielded after treatment with soda 0.0882 gm.  $\text{Yb}_2\text{O}_3$  and 0.6290 gm.  $\text{WO}_3$ .

|                            | Calculated for<br>$\text{Yb}_2\text{O}_3 \cdot 12\text{WO}_3 + 35\text{H}_2\text{O}$<br>in per cent. |       | Found. |       |
|----------------------------|------------------------------------------------------------------------------------------------------|-------|--------|-------|
|                            |                                                                                                      |       | I.     | II.   |
| $\text{Yb}_2\text{O}_3$ .. | 394.2 10.35                                                                                          | 83.44 | 83.86  | 10.29 |
| $12\text{WO}_3$ ..         | 2784. 73.09                                                                                          |       |        | 73.39 |
| $35\text{H}_2\text{O}$ ..  | 630.7 16.56                                                                                          |       |        | 16.29 |
|                            | 3808.9 100.00                                                                                        |       |        | 99.97 |

A completely analogous samarium salt,—



has been prepared by Cleve.

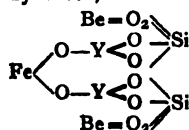
Ytterbium Oxytungstate,  $\text{Yb}_2\text{O}_3 \cdot \text{WO}_3 = (\text{YbO})_2 \cdot \text{WO}_4$ .

If equivalent quantities of ytterbium oxide and tungstic acid anhydride are ignited in a flux of common salt, only very little action takes place. It is better to act on the earth in presence of larger quantities of  $\text{WO}_3$ , but even under these conditions it remains for the most part undissolved. After cooling, the residue is powdered and again ignited with a similar flux. The part that still remains undissolved, after washing with water, was seen to be a mixture of two crystalline salts—a lighter colourless salt, which was separated by washing from the other heavier glittering compact powder, of a faintly reddish grey colour. The latter appeared under the microscope to consist of perfectly-developed short and thick hexagonal prisms with rectangular sections, and sometimes base pyramids. These prisms were analysed.

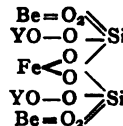
0.8184 gm. of the finely-powdered substance, after protracted treatment with concentrated hydrochloric acid, gave 0.3007 gm.  $\text{WO}_3$ , 0.5146 gm.  $\text{Yb}_2\text{O}_3$  was precipitated in the filtrate by ammonia.

|                            | Calculated for<br>$\text{Yb}_2\text{O}_3 \cdot \text{WO}_3$ , in per cent. | Found. |
|----------------------------|----------------------------------------------------------------------------|--------|
| $\text{Yb}_2\text{O}_3$ .. | 394.2 62.95                                                                | 62.88  |
| $\text{WO}_3$ ..           | 232. 37.05                                                                 | 36.74  |
|                            | 626.2 100.00                                                               | 99.62  |

The salt represents a type hitherto unknown in the case of the rare earths,  $\text{R}^{\text{III}}\text{O}-\text{O} > \text{R}^{\text{III}}\text{O}_2$ ; where  $\text{R}^{\text{III}}\text{O}$  is a monovalent positive radical, which is of interest inasmuch as it affords an experimental support for the latest formula of gadolinite. The improbability of the older formula suggested by Groth,—



was demonstrated by W. Pettersson ("Studier öfver Gadolinit," *Geol. Förr. Förh.*, Bd. 12, Häft 4, p. 70, Stockholm), and this author put forward the following formula:—



Thus, on the assumption that this last formula, which is in any case preferable, is correct, the rare earths occur in nature as the radical  $\text{R}^{\text{III}}\text{O}$ .

Sodium-ytterbium Tungstates.

1.  $2\text{Yb}_2\text{O}_3 \cdot 4\text{Na}_2\text{O} \cdot 7\text{WO}_3$ .

This salt was formed at the same time as the preceding, and was obtained from the mixture by washing from the heavier portion. A small quantity also crystallised from the fused mass on cooling. Under the microscope the crystals exhibited a central stem and were developed in two opposite directions in needles with pointed ends. The material, rendered as homogeneous and uniform as possible, was analysed:—

0.4552 gm. of the substance gave 0.1340 gm.  $\text{Yb}_2\text{O}_3$  and 0.2727 gm.  $\text{WO}_3$ . From the earth precipitated from the filtrate by ammonia 0.0910 gm.  $\text{NaCl}$  was obtained.

|                             | Calculated for<br>$2\text{Yb}_2\text{O}_3 \cdot 4\text{Na}_2\text{O} \cdot 7\text{WO}_3$<br>in per cent. | Found. |
|-----------------------------|----------------------------------------------------------------------------------------------------------|--------|
| $2\text{Yb}_2\text{O}_3$ .. | 788.4 29.63                                                                                              | 29.44  |
| $4\text{Na}_2\text{O}$ ..   | 248.4 9.34                                                                                               | 10.61  |
| $7\text{WO}_3$ ..           | 1624. 61.03                                                                                              | 59.91  |
|                             | 2660.8 100.00                                                                                            | 99.96  |

The fact that too high a percentage of sodium was found, and a correspondingly too low percentage of tungstic acid, was due to the difficulty in separating these two substances.

2.  $\text{Yb}_2\text{O}_3 \cdot 9\text{Na}_2\text{O} \cdot 12\text{WO}_3$ .

This is the probable composition of another double salt which is obtained in small quantity when nearly pure sodium chloride is used as a flux. It forms opaque microscopic needles, insoluble in water.

Analyses.

0.1037 gm. of the substance, treated as above, gave 0.0102 gm.  $\text{Yb}_2\text{O}_3$ , 0.0750 gm.  $\text{WO}_3$ , and 0.0283 gm.  $\text{NaCl}$ .

|                            | Calculated for<br>$\text{Yb}_2\text{O}_3 \cdot 9\text{Na}_2\text{O} \cdot 12\text{WO}_3$<br>in per cent. | Found. | Quotients. |
|----------------------------|----------------------------------------------------------------------------------------------------------|--------|------------|
| $\text{Yb}_2\text{O}_3$ .. | 394.2 10.55                                                                                              | 9.8    | 1          |
| $9\text{Na}_2\text{O}$ ..  | 558.9 14.95                                                                                              | 14.5   | 9.2        |
| $12\text{WO}_3$ ..         | 2784. 74.50                                                                                              | 72.3   | 12.5       |
|                            | 3737.1 100.00                                                                                            | 96.6   |            |

No control analysis could be performed for want of sufficient material. But the numbers found are too low, probably owing to the presence of impurities, as the quotients agree fairly well with the formula suggested.

The double sodium tungstates of the rare earths have been specially examined by Högbom (*Öf. K. Vet. Ak. Förh.*, Stockholm, 1884, No. 5, p. 111). But attempts to prepare the pure products have as yet been unsuccessful in the case of ytterbium. Neither of the above salts, moreover, belongs to the types put forward by Högbom, which all contain equivalent quantities of acid and basic anhydride. Similarly in the case of the double salt described last, which, on the other hand, is richer in sodium than all hitherto known corresponding compounds in the yttrium group, and in that respect most closely resembles sodium-thorium tungstate,  $\text{ThO}_2 \cdot 14\text{Na}_2\text{O} \cdot 16\text{WO}_3$  (Högbom, *loc. cit.*, p. 118). With excess of metallic oxide,

\* From the *Zeitschrift für Anorganische Chemie*, xxxii.

Salt 1 forms the transition to the type of the ytterbium oxytungstate.

**Ytterbium Formate,  $\text{Yb}_3\text{CHO}_2 + 2\text{H}_2\text{O}$**   
(Dried over Sulphuric Acid).

Forms little needles which unite into hard opaque little balls; very soluble. All water of crystallisation passes off at  $100^\circ$ . A very voluminous oxide remains behind after ignition.

**Analyses.**

1. 1.1270 grms. of the salt dried over sulphuric acid lost 0.1205 grm. at  $130^\circ$ .
2. 0.7025 grm. of the salt dried over sulphuric acid lost 0.0786 grm. at  $100^\circ$ , and after ignition yielded 0.4000 grm.  $\text{Yb}_2\text{O}_3$ .

|                          | Calculated for<br>$\text{Yb}_3\text{CHO}_2 + 2\text{H}_2\text{O}$ ,<br>in per cent. |       | Found. |       |
|--------------------------|-------------------------------------------------------------------------------------|-------|--------|-------|
|                          | I.                                                                                  | II.   | I.     | II.   |
| Yb .. ..                 | 173.1                                                                               | 50.30 | —      | 50.02 |
| $3\text{CHO}_2$ ..       | 135.03                                                                              | 39.23 | —      | —     |
| $2\text{H}_2\text{O}$ .. | 36.04                                                                               | 10.47 | 10.69  | 11.19 |

344.17 100.00

3. 1.2117 grm. of the pressed substance lost at  $130^\circ$  0.2047 grm., equals 16.84 per cent. For  $3\frac{1}{2}\text{H}_2\text{O}$  the calculated value is 16.99 per cent.

**Ytterbium Acetate,  $\text{Yb}_3\text{C}_2\text{H}_3\text{O}_2 + 4\text{H}_2\text{O}$ .**

As ytterbium oxide is not soluble in glacial acetic acid, the salt was prepared from the hydrate. It forms small six-sided stable tablets, readily soluble in water. It has a feebly alkaline reaction in solution. At  $100^\circ$  all the water of crystallisation passes off.

**Analysis of the Pressed Salt.**

- 0.8271 grm. of the substance lost 0.1427 grm. at  $110^\circ$ , and yielded after ignition 0.3829 grm.  $\text{Yb}_2\text{O}_3$ .

|                                      | Calculated for<br>$\text{Yb}_3\text{C}_2\text{H}_3\text{O}_2 + 4\text{H}_2\text{O}$ ,<br>in per cent. |       | Found. |
|--------------------------------------|-------------------------------------------------------------------------------------------------------|-------|--------|
|                                      | I.                                                                                                    | II.   |        |
| Yb .. ..                             | 173.1                                                                                                 | 40.99 | 40.66  |
| $3\text{C}_2\text{H}_3\text{O}_2$ .. | 179.09                                                                                                | 41.94 | —      |
| $4\text{H}_2\text{O}$ ..             | 72.08                                                                                                 | 17.07 | 17.25  |

422.27 100.00

Specific gravity .. .. 2.09  
Molecular volume .. .. 202.

**Ytterbium Propionate.**

1.  $\text{Yb}_3\text{C}_3\text{H}_5\text{O}_2 + 3\text{H}_2\text{O}$ .

Obtained at the temperature of the room in greasy-feeling scales forming spherical aggregations. It gives up no water over sulphuric acid.

**Analysis of the Pressed Salt.**

- 1.0724 grm. of the substance gave 0.4679 grm.  $\text{Yb}_2\text{O}_3$ .

|          | Calculated for<br>$\text{Yb}_3\text{C}_3\text{H}_5\text{O}_2 + 3\text{H}_2\text{O}$ ,<br>in per cent. |       | Found. |
|----------|-------------------------------------------------------------------------------------------------------|-------|--------|
|          | I.                                                                                                    | II.   |        |
| Yb .. .. | 38.79                                                                                                 | 38.32 | —      |

2.  $\text{Yb}_3\text{C}_3\text{H}_5\text{O}_2 + \text{H}_2\text{O}$ .

Similar to the former, but separates out at the temperature of water.

**Analysis of the Pressed Salt.**

- 1.0447 grm. of the substance gave on combustion 0.3620 grm.  $\text{H}_2\text{O}$ , 1.0080 grm.  $\text{CO}_2$ , and 0.5054 grm.  $\text{Yb}_2\text{O}_3$ .

|                       | Calculated for<br>$\text{Yb}_3\text{C}_3\text{H}_5\text{O}_2 + \text{H}_2\text{O}$ ,<br>in per cent. |       | Found. |
|-----------------------|------------------------------------------------------------------------------------------------------|-------|--------|
|                       | I.                                                                                                   | II.   |        |
| Yb .. ..              | 173.1                                                                                                | 42.19 | 42.49  |
| $\text{C}_3$ .. ..    | 108.                                                                                                 | 26.32 | 25.85  |
| $\text{H}_{17}$ .. .. | 17.17                                                                                                | 4.18  | 3.85   |
| $\text{O}_7$ .. ..    | 112.                                                                                                 | 27.41 | —      |

410.27 100.00

**Ytterbium Oxalate,  $\text{Yb}_2\text{C}_2\text{O}_4 + 10\text{H}_2\text{O}$ .**

This salt has been described by Nilson (*Ofv. Vet. Ak. Förh., Stockholm*, 1880, No. 6, p. 11). I need only add some determinations of the solubility and specific gravity.

**Specific Gravity and Molecular Volume.**

- (a). Dried in air ( $10\text{H}_2\text{O}$ ): 1.0769 grm. Specific gravity = 2.644; molecular volume, 299.
- (b). Dried over sulphuric acid: 1.0907 grm. Specific gravity = 2.439; molecular volume, 309.

**Solubility of the Oxalate in Ammonium Oxalate**

By an experiment carried out according to Brauner's directions ("Contribution to the Chemistry of Thorium," *Trans. Chem. Soc.*, 1898, p. 951), it was proved that 1.2903 grm. crystallised ammonium oxalate (= 1 grm. anhydrous) dissolved in 38.265 grm. of water held in solution 0.02437 grm.  $\text{Yb}_2\text{O}_3$  at the temperature of the room. Thus, the solubility is about ten times as great as that of yttrium oxalate, for which the corresponding value, according to Brauner, amounts to 0.002562 grm. ( $\text{Y}_2\text{O}_3$ ).

**Solubility in Normal Sulphuric Acid.**

- (a). 100 c.c. normal sulphuric acid were boiled for two hours with excess of ytterbium oxalate. After cooling, the solution required—

|                                                                                                                     | $\text{Yb}_2\text{C}_2\text{O}_4$<br>Grm. |
|---------------------------------------------------------------------------------------------------------------------|-------------------------------------------|
| 1. 31.71 c.c. of a chameleon solution equivalent to 0.007424 grm. ammonium oxalate per c.c., corresponding to .. .. | 0.3857                                    |
| 2. 30.11 c.c. of the same solution, corresponding to .. ..                                                          | 0.3581                                    |
| Mean .. ..                                                                                                          | 0.3719                                    |

- (b). 100 c.c. normal sulphuric acid required after repeated shaking in the cold with excess of ytterbium oxalate 25.09 c.c. of the same permanganate solution corresponding to 0.3053 grm.  $\text{Yb}_2\text{C}_2\text{O}_4$ .

Hence it follows that, in sulphuric acid also, the solubility is comparatively great, e.g., three to four times as great as that of gadolinium oxalate and about twice as great as that of yttrium oxalate.

**Acid Ytterbium Malonate,  $\text{YbH}_2\text{C}_2\text{H}_2\text{O}_4$ .**

Ytterbium hydrate was first dissolved by excess of malonic acid, but the solution soon deposited the above acid salt in fine needles, which were soluble only with great difficulty. The salt after pressing between paper is anhydrous.

**Analyses.**

1. 0.1548 grm. of the substance dried in the desiccator gave on combustion 0.1078 grm.  $\text{CO}_2$ , 0.0181 grm.  $\text{H}_2\text{O}$ , and 0.0812 grm.  $\text{Yb}_2\text{O}_3$ .
2. 0.2616 grm. of the pressed substance gave 0.1355 grm.  $\text{Yb}_2\text{O}_3$ .

|                    | Calculated for<br>$\text{YbH}_2\text{C}_2\text{H}_2\text{O}_4$ ,<br>in per cent. |       | Found. |       |
|--------------------|----------------------------------------------------------------------------------|-------|--------|-------|
|                    | I.                                                                               | II.   | I.     | II.   |
| Yb .. ..           | 173.1                                                                            | 45.78 | 46.06  | 45.49 |
| $\text{C}_6$ .. .. | 72.                                                                              | 19.04 | 18.99  | —     |
| $\text{H}_5$ .. .. | 5.05                                                                             | 1.33  | 1.30   | —     |
| $\text{O}_8$ .. .. | 128.                                                                             | 33.85 | —      | —     |

378.15 100.00

**Ytterbium Succinate,  $\text{Yb}_2\text{C}_4\text{H}_4\text{O}_4 + 3\text{H}_2\text{O}$ .**

Succinic acid gives no precipitate with solutions of ytterbium acetate in the cold. If the solution is boiled for a short time, the granular succinate is precipitated. It is soluble only with great difficulty.

**Analyses of the Salt Dried in the Desiccator.**

- 0.3341 grm. of the substance gave on combustion 0.2368 grm.  $\text{CO}_2$ , 0.0690 grm.  $\text{H}_2\text{O}$ , and 0.1758 grm.  $\text{Yb}_2\text{O}_3$ .

|                       | Calculated for<br>$\text{Yb}_2\text{C}_6\text{H}_5\text{O}_7 + 3\text{H}_2\text{O}$ ,<br>in per cent. | Found. |
|-----------------------|-------------------------------------------------------------------------------------------------------|--------|
| Yb <sub>2</sub> .. .. | 346.2                                                                                                 | 46.20  |
| C <sub>12</sub> .. .. | 144                                                                                                   | 19.33  |
| H <sub>18</sub> .. .. | 18.18                                                                                                 | 2.43   |
| O <sub>15</sub> .. .. | 240                                                                                                   | 32.07  |
|                       | 748.38                                                                                                | 100.00 |

*Ytterbium Lactate,  $\text{Yb}_2\text{C}_3\text{H}_5\text{O}_3 + 2\text{H}_2\text{O}$ .  
(Dried over Sulphuric Acid).*

The addition of lactic acid to ytterbium acetate in the cold caused no turbidity; if the solution is heated, a gelatinous precipitate of the above salt is formed, and may be pressed between paper.

*Analysis of the Salt Dried over Sulphuric Acid.*

- 0.9128 grm. of the substance suffered a loss in weight of 0.0175 grm. at 100°, equals 1.92 per cent. 0.6444 grm. of the residue gave 0.2684 grm.  $\text{Yb}_2\text{O}_3$ .
- 0.2949 grm. of the substance gave on combustion 0.2505 grm.  $\text{CO}_2$ , 0.1045 grm.  $\text{H}_2\text{O}$ , and 0.1203 grm.  $\text{Yb}_2\text{O}_3$ .

|                       | Calculated for<br>$\text{Yb}_2\text{C}_3\text{H}_5\text{O}_3 + 2\text{H}_2\text{O}$ , in per cent. | Found.      |
|-----------------------|----------------------------------------------------------------------------------------------------|-------------|
|                       |                                                                                                    | I. II.      |
| Yb .. ..              | 173.1                                                                                              | 35.88 35.82 |
| C <sub>9</sub> .. ..  | 108                                                                                                | — 23.17     |
| H <sub>19</sub> .. .. | 19.19                                                                                              | — 3.94      |
| O <sub>11</sub> .. .. | 176                                                                                                | — —         |
|                       | 476.29                                                                                             |             |

*Acid Ytterbium Tartrates.*

*1.  $\text{YbH}_2\text{C}_4\text{H}_4\text{O}_6 + 12\text{H}_2\text{O}$ .*

Tartaric acid gives with cold solutions of ytterbium acetate a flocculent precipitate, which, on warming with excess of the acid, again goes into solution. If the tartaric acid is added in exactly sufficient quantity, the acid tartrate crystallises on cooling in needles, which only dissolve with difficulty. A small quantity was crystallised from water and analysed.

- 0.9868 grm. of the pressed salt gave up 0.1366 grm. of water in the desiccator, and 0.1631 grm. more at 110°. 0.6771 of the residue gave 0.2777 grm.  $\text{Yb}_2\text{O}_3$ .
- 0.3248 grm. of the salt dried over sulphuric acid gave 0.1077 grm.  $\text{Yb}_2\text{O}_3$ .

|          | Calculated for<br>$\text{YbH}_2\text{C}_4\text{H}_4\text{O}_6 + 7\text{H}_2\text{O}$ ,<br>in per cent. | Found.      |
|----------|--------------------------------------------------------------------------------------------------------|-------------|
|          |                                                                                                        | I. II.      |
| Yb .. .. | 29.03                                                                                                  | 29.11 29.12 |

|                         | Calculated for<br>$\text{YbH}_2\text{C}_4\text{H}_4\text{O}_6 + 12\text{H}_2\text{O}$ ,<br>in per cent. | Found.                                   |
|-------------------------|---------------------------------------------------------------------------------------------------------|------------------------------------------|
| 5H <sub>2</sub> O .. .. | 13.13                                                                                                   | 13.84 (loss of wt. over sulphuric acid). |

*2.  $\text{YbH}_2\text{C}_4\text{H}_4\text{O}_6 + 2\text{H}_2\text{O}$  (over Sulphuric Acid).*

After being kept for some time, a partially crystallised preparation had this composition.

*Analysis.*

0.7590 grm. of the substance gave on combustion 0.5213 grm.  $\text{CO}_2$ , 0.1697 grm.  $\text{H}_2\text{O}$ , and 0.2922 grm.  $\text{Yb}_2\text{O}_3$ .

|                       | Calculated for<br>$\text{YbH}_2\text{C}_4\text{H}_4\text{O}_6 + 2\text{H}_2\text{O}$ , in per cent. | Found. |
|-----------------------|-----------------------------------------------------------------------------------------------------|--------|
| Yb .. ..              | 173.1                                                                                               | 33.81  |
| C <sub>8</sub> .. ..  | 96                                                                                                  | 18.96  |
| H <sub>13</sub> .. .. | 13.13                                                                                               | 2.59   |
| O <sub>14</sub> .. .. | 224                                                                                                 | 44.26  |
|                       | 506.23                                                                                              | 100.00 |

A similar acid yttrium salt ( $3\text{H}_2\text{O}$ ) has been prepared by Cleve.

*Acid Ytterbium Citrates.*

*1.  $2\text{YbC}_6\text{H}_5\text{O}_7 + \text{H}_3\text{C}_6\text{H}_5\text{O}_7 + 12\text{H}_2\text{O}$ .*

This salt is obtained from ytterbium hydroxide by the addition of just sufficient citric acid to dissolve it. It crystallises from concentrated solutions in little needles which collect together in rays. When dried at 100° the salt is anhydrous.

*Analysis.*

- 0.6277 grm. of the pressed substance gave up 0.1098 grm. of water in the desiccator; at 110°, 0.0057 grm. more; thus, 0.1155 grm. in all, corresponding to 18.32 per cent. In the given formula are 12H<sub>2</sub>O, equals 19.09 per cent.
- 0.4492 grm. of the substance dried at 100° gave 0.1948 grm.  $\text{Yb}_2\text{O}_3$ .
- 0.5122 grm. of the substance dried at 100° gave 0.2221 grm.  $\text{Yb}_2\text{O}_3$ .

|                               | Calculated for<br>$2\text{YbC}_6\text{H}_5\text{O}_7 + \text{H}_3\text{C}_6\text{H}_5\text{O}_7$ ,<br>in per cent. | Found.      |
|-------------------------------|--------------------------------------------------------------------------------------------------------------------|-------------|
|                               |                                                                                                                    | I. II.      |
| $\text{Yb}_2\text{O}_3$ .. .. | 37.78                                                                                                              | 38.08 38.08 |

*2.  $2\text{YbC}_6\text{H}_5\text{O}_7 + \text{H}_3\text{C}_6\text{H}_5\text{O}_7 + 15\text{H}_2\text{O}$ .*

This formula gives the amount of water combined when the substance is kept in an atmosphere saturated with moisture.

*Analysis of the Pressed Salt.*

0.8907 grm. of the substance gave up 0.1994 grm. of water at 100°, equals 22.37 per cent. According to the above formula, the percentage of water calculated for 15H<sub>2</sub>O is 22.77 per cent.

*Ytterbium Benzoate,  $\text{Yb}_2\text{C}_7\text{H}_5\text{O}_2$ .*

Results as a granular precipitate on bringing together solutions of ytterbium acetate and benzoic acid. It is soluble with very great difficulty in boiling water. The salt is anhydrous, and can be heated to 130° without loss of weight.

*Analysis of the Crystallised Salt.*

0.2405 grm. of the substance gave 0.0890 grm.  $\text{Yb}_2\text{O}_3$ .

|          | Calculated for<br>$\text{Yb}_2\text{C}_7\text{H}_5\text{O}_2$ , in per cent. | Found. |
|----------|------------------------------------------------------------------------------|--------|
| Yb .. .. | 32.28                                                                        | 32.50  |

*Ytterbium Picrate,  $\text{Yb}_2\text{C}_6\text{H}_2(\text{NO}_2)_3\text{O} + 8\text{H}_2\text{O}$ .*

It forms long perfectly developed columns. The salt is readily soluble, slowly effloresces in the air, and dissolves at about 80° in its water of crystallisation. On heating more strongly, it explodes. Half of the water passes off over sulphuric acid, and the rest at 100°.

*Analysis.*

- 2925 grm. of the pressed substance gave off 0.0434 grm. of water at 100°.
- 0.3643 grm. of the pressed substance gave up 0.0274 grm. of water over sulphuric acid, and 0.0272 grm. more at 100°.
- 0.2714 grm. of the salt dried at 100° gave, on precipitation with ammonia, 0.0611 grm.  $\text{Yb}_2\text{O}_3$ .

|                                                              | Calculated for<br>$\text{Yb}_2\text{C}_6\text{H}_2(\text{NO}_2)_3\text{O} + 8\text{H}_2\text{O}$ ,<br>in per cent. | Found.                  |
|--------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------|-------------------------|
|                                                              |                                                                                                                    | I. II. III.             |
| Yb .. ..                                                     | 173.1                                                                                                              | 17.28 — — 16.84         |
| 3C <sub>6</sub> H <sub>2</sub> N <sub>3</sub> O <sub>7</sub> | 684.42                                                                                                             | 68.32 — — —             |
| 4H <sub>2</sub> O .. ..                                      | 72.08                                                                                                              | 7.20 14.40 14.84 7.52 — |
| 4H <sub>2</sub> O .. ..                                      | 72.08                                                                                                              | 7.20 — — 7.47 —         |
|                                                              | 1001.68                                                                                                            | 100.00                  |

*Specific Gravity and Molecular Volume.*—0.3238 grm. substance. Specific gravity, 1.95; Molecular volume, 513.

(To be continued).

## PROCEEDINGS OF SOCIETIES.

## CHEMICAL SOCIETY.

Ordinary Meeting, Thursday, December 4th, 1902.

Dr. W. H. PERKIN, F.R.S., Vice-President, in the Chair.

Mr. J. H. RYFFEL was formally admitted a Fellow of the Society.

Certificates were read for the first time in favour of Messrs. Francis George Cousins, Queen's Road, London, N.; Archibald Sefton Elford, B.A., 9, Keble Road, Oxford; Moungh Tha Huyin, B.A., 18, Mercers Road, London, N.; Maurice Kemp-Welsh, Parkstone, Weybridge, Surrey; Samuel Edward Sibley, 3, Rutland Road, Ilford; Duncan Turner, Elmbank, Broomhouse, near Glasgow.

A ballot for the election of Fellows was held, when the following were declared duly elected:—John Frederick Alder; Alfred Edward Beanes; Herbert Blair; John A. H. Brincker, B.A., M.B.; Vernon Seymour Bryant, B.A.; Thomas Burnell Carmichael; Charles Cockle; James Justinian Drought; Edgar Leeder Edlin, B.A.; Sidney Robert Edminson, B.Sc.; Walter Henry Edwards; George Felix Dudbridge Green; Wilfrid Russell Grimwade, B.Sc.; Edward Gumersall; David Vincent Hollingworth; Alfred Holt, jun., B.A.; Arthur Francis Hoaking; Clements F. V. Jackson; Douglas Kennedy Jardine, B.Sc.; John Petty Leather; Frank Austin Lidbury, M.Sc.; Ernest Liotard; Thomas Stratford Logan; Douglas A. MacCallum; William Mann, B.Sc.; John Marsh; Joseph William Mellor, D.Sc.; John Price Millington, B.A., B.Sc.; Beni Madhav Mukerjee; John Phelps, B.A.; Richard Pribram, Ph.D.; Guy Dunstan Ricketts, M.A.; George Archibald Park Ross, M.B.; John William Sampson; Duncan Simpson; Robert Walter Sindall; Leonard Smith; John Seabury Smythe, B.Sc., Ph.D.; Edgar Stansfield, B.Sc.; Walter F. Sutherland, Ph.D.; George Sutherland Thomson; Henry Lethby Tidy, B.A.; Thomas Edward Wallis, B.Sc.; William Carter White; Lyndon Wilson.

Of the following papers, those marked \* were read:—

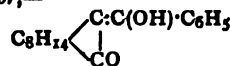
\*173. "The Specific Heats of Liquids." By H. CROMPTON.

It is assumed that when heat is applied to a liquid, if there is no change in its molecular composition, (1) there is an increase in the kinetic energy of the molecules, (2) internal work is done within each molecule, (3) there is a diminution in the attraction of the molecules for one another, (4) some external work is done. The last quantity is neglected in considering the specific heat, and the first and second changes are taken to have the same effect on the specific heat of the liquid and on that of its vapour, so that together they make up the specific heat at constant volume of the vapour. At any absolute temperature  $T$ , the molecular heat at constant volume of the vapour  $C_v = 2.06 + 0.00245 M \nu_b T \log n$ , where  $n$  is the number of atoms in the molecule, and  $M \nu_b$  is the molecular volume of the liquid at the boiling-point. The molecular attraction is regarded as measured by the difference between the latent heat of condensation of the vapour and the heat evolved on simply compressing the vapour into the volume of the liquid without causing a change of state. This attraction is assumed to become zero at the critical temperature, and to increase regularly with the fall in temperature from the critical point downwards. At the absolute temperature  $T$ , the change in the attraction is therefore  $dA/dT = (L - RT \log V_0/v_0)/(T_k - T)$ , in which  $L$  is the latent heat of condensation,  $R$  the constant of the gas equation,  $V_0$  and  $v_0$  the volumes of the vapour and liquid respectively, and  $T_k$  the absolute critical temperature. It follows from the above that the molecular heat of the liquid is  $C_l = C_v + dA/dT$ , and the author showed that the molecular heats of liquids thus calculated

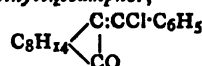
agree fairly with those observed, except where molecular association occurs in the liquid.

\*174. "The Constitution of Enolic Benzoylcamphor." By M. O. FORSTER.

From the production of camphorquinone by oxidising benzoylcamphor with chromic acid and with mercuric acetate, it is concluded that the enolic modification of benzoylcamphor has the constitution of *phenylhydroxy-methylencamphor*,—



Phenylchloromethylencamphor,—



obtained from enolic benzoylcamphor and phosphorus pentachloride, melts at  $100^\circ$  and its rotation  $[\alpha]_D = -217^\circ$ ; the alcoholic solution gives no colouration with ferric chloride. Aniline converts it into *anilinophenylmethylencamphor anil*,  $\text{C}_{29}\text{H}_{30}\text{N}_2$ , sulphur-yellow plates, m. p.  $117-118^\circ$ ; the *acetyl* derivative,  $\text{C}_{31}\text{H}_{32}\text{ON}_2$ , melts at  $166^\circ$ .

When phenylchloromethylencamphor is heated with alcoholic ammonia in sealed tubes at  $150-170^\circ$ , a *base* having the formula  $\text{C}_{17}\text{H}_{21}\text{ON}$  is produced. This melts at  $170^\circ$ , its rotation  $[\alpha]_D = +180.2^\circ$ ; it gives an intense blue colouration with ethereal ferric chloride; the *picrate* melts at  $157^\circ$ , and the *benzoyl* derivative at  $99-100^\circ$ .

On heating enolic benzoylcamphor with ammonium formate in sealed tubes at  $200-220^\circ$ , another *base*, also having the formula  $\text{C}_{17}\text{H}_{21}\text{ON}$ , is formed. It melts at  $118-119^\circ$ , its rotation  $[\alpha]_D = +235.2^\circ$ ; it gives no colour with ferric chloride; it does not combine with benzaldehyde or benzoyl chloride, but yields a *picrate* which does not melt at  $250^\circ$ , and a *bromo-derivative*,  $\text{C}_{17}\text{H}_{20}\text{ONBr}$ , orange-red plates, m. p.  $173^\circ$ .

Enolic benzoylcamphor is reduced in alkaline solution by sodium amalgam, yielding benzylidenecamphor and benzoylcamphor. Potassium permanganate oxidises it to a mixture of benzoic and camphoric acids, whilst potassium ferricyanide gives rise to a *compound* having the formula  $\text{C}_{34}\text{H}_{38}\text{O}_4$  and m. p.  $221^\circ$ , its rotation  $[\alpha]_D = +270.4^\circ$ ; it does not react with ferric chloride, and is insoluble in alkalis.

\*175. "Isomeric Benzoyl Derivatives from isoNitrosocamphor." By M. O. FORSTER.

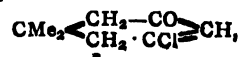
When *isonitrosocamphor* is treated with benzoyl chloride in presence of sodium hydroxide, two compounds are produced each having the empirical formula  $\text{C}_{17}\text{H}_{19}\text{O}_3\text{N}$ .

The *compound* from which *isonitrosocamphor* can be regenerated by hydrolysis, crystallises from alcohol in large, yellow prisms melting at  $105-106^\circ$ ; it is readily soluble in petroleum, and its rotation  $[\alpha]_D = +145.6^\circ$  in chloroform. The *isonitrosocamphor* obtained from this derivative has the rotation  $[\alpha]_D = +221.9^\circ$ , and yields the two new substances on benzoylation.

The *compound* which accompanies the above-mentioned derivative, although isomeric with it, does not give rise to *isonitrosocamphor* on hydrolysis; it is scarcely soluble in petroleum, and crystallises from alcohol in colourless, lustrous laminæ melting at  $136^\circ$ , and its rotation  $[\alpha]_D = +126.9^\circ$  in chloroform. Alcoholic potash resolves it into a mixture of benzoic and cyanolauronic (*α-camphornitrilic*) acids.

\*176. "Action of Phosphorus Haloids on Dihydroresorcins. Part I. Dimethyldihydroresorcin." By A. W. CROSSLEY and H. R. LE SUEUR.

Phosphorus trichloride reacts with dimethyldihydroresorcin, giving 5-chloro-3-keto-1 : 1-dimethyl-Δ<sup>4</sup>-tetrahydrobenzene,—





a colourless liquid boiling at  $109^{\circ}$  (14 m.m.), and also small quantities of an anhydride of dimethyldihydroresorcin,  $(C_6H_{11}O)_2O$ , melting at  $99.5^{\circ}$ .

Phosphorus oxychloride gives the same chloroketone, and phosphorus tribromide the corresponding bromo-compound,  $C_6H_{11}OBr$ , which boils at  $129^{\circ}$  (25 m.m.).

When phosphorus pentachloride acts on dimethyldihydroresorcin, 3:5-dichloro-1:1-dimethyl- $\Delta$  2:4-dihydrobenzene (*Trans.*, 1902, lxxxi., 826) and 3:5-dichloro-o-xylene (*Trans.*, 1902, lxxxi., 1533) are obtained.

By the action of phosphorus pentabromide on dimethyldihydroresorcin, bromodimethyldihydroresorcin is first formed, and the products of the action of the phosphorus bromides on this compound have been investigated. With phosphorus tribromide there is obtained a mixture of mono- and di-bromoketodimethyltetrahydrobenzene, the latter, having the formula  $C_8H_{10}OBr_2$ , melts at  $96^{\circ}$ ; and a liquid which on treatment with bromine is converted into a tribromoxylene which melts at  $176-177.5^{\circ}$ . By the further action of bromine the above dibromoketone is converted into tri- and tetra-bromoketodimethyltetrahydrobenzene, which melt respectively at  $106^{\circ}$  and  $118^{\circ}$ . These latter substances are also produced by the action of phosphorus pentabromide on bromodimethyldihydroresorcin.

The action of phosphorus pentabromide on dimethyldihydroresorcin is one which is largely influenced by the conditions under which the experiments are carried out. The following products of the reaction have been isolated:—Bromodimethyldihydroresorcin, tribromoketotetrahydrobenzene, a bromoxylene,  $C_8H_9OBr$ , melting at  $83.5-84^{\circ}$ , a dibromoxylene melting at  $96.5^{\circ}$ , and two tribromoxylens melting at  $176-177.5^{\circ}$  and  $182-183^{\circ}$  respectively.

177. "The Absorption Spectra of Metallic Nitrates." Part II. By W. N. HARTLEY, D.Sc., F.R.S.

The substances examined were aqueous solutions of the nitrates of fifteen metals, and of nitric, sulphuric, and hydrochloric acids, and alcoholic solutions of ethyl and lithium nitrates.

Metals which exhibit characteristic absorption bands show variations in the positions and intensities of these bands which appear to be dependent upon the molecular weights of the salts in solution. Equivalent quantities of certain metals exhibit the same absorption spectra whether in strong or dilute solution, and, in certain cases, through a great range in dilution. A dilute solution of silver nitrate transmits a longer range of spectrum than a strong solution containing the same quantity of salt. The effect of dilution on nitric acid of sp. gr. 1.42 is the reverse of that observed in the case of silver nitrate. There is a remarkable difference between the constitution of ethyl nitrate on the one hand (whether undiluted or in dilute alcoholic solution), and that of solutions equally dilute of inorganic nitrates on the other. The effect of raising the temperature of the solutions is simply to intensify the absorption spectrum as if a stronger solution at a lower temperature had been used.

Although evidence of what may be regarded as ionic dissociation is afforded by the spectra of dilute solutions of the inorganic nitrates, yet the spectra themselves are not alike, as they would be if the salts were completely resolved into  $NO_3$  and metallic ions; the evidence is just as favourable to the view that some of the salts are resolved into nitric acid and the corresponding base.

In the case of esters, like ethyl nitrate, the conditions are entirely different, as the molecule is clearly not dissociated, there being no evidence of the  $-NO_3$  group in the compound.

Views in explanation of the facts observed were put forward.

178. "The Constitution of the Products of Nitration of Meta-acetoluidide." By J. B. COHEN and H. D. DAKIN.

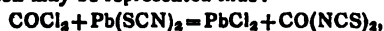
The authors show that the nitro-derivative of meta-acetoluidide obtained by Bellstein and Kuhlberg (*Annalen*, 1871, clviii., 348) contains the nitro-group in the para-

position with respect to the amido-group, and not in the ortho-position as stated by Limpricht (*Ber.*, 1885, xviii., 403). The substance was successively converted into 3-chloro-6-nitrotoluene and 3-chloro-4:6-dinitrotoluene.

By fractional crystallisation from alcohol of the products of nitration, a nitro-derivative which had hitherto escaped observation was separated, which proved to be 4-nitro-3-acetylaminotoluene. On hydrolysis it gave a base identical with the 4-nitro-3-aminotoluene obtained by Staedel and Kolb (*Annalen*, 1890, cclix., 224) by the action of ammonia on 4-nitro-*m*-cresol ethyl ether.

179. "The Action of Metallic Thiocyanates upon Carbonyl Chloride." By A. E. DIXON.

When carbonyl chloride dissolved in toluene is allowed to stand in contact with excess of finely divided sodium, potassium, barium, mercury, or lead thiocyanate, interaction gradually occurs with formation of metallic chloride, and a substance possessing the general chemical characters of a thiocarbimide, but giving, under certain conditions, the reactions of a thiocyanate. It has recently been pointed out by the author that tautomerism of this kind often occurs among the so-called "thiocyanates" of electro-negative radicles (*Trans.*, 1901, lxxix., 541). The interaction may be represented thus:—

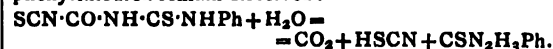


but it does not take place quantitatively, isopropylphosphanic acid and pseudo-sulphocyanogen and occasionally other products being formed. Heating to about  $100^{\circ}$  greatly accelerates the change, but even after thirty hours not more than one-third of the carbonyl chloride employed is converted into thiocarbimide, and much remains unchanged.

Hitherto, all attempts to isolate carbonyldithiocarbimide in a pure state have failed, but its solution interacts with alcohol and with nitrogenous bases, yielding additive products.

Only one molecule of ethyl alcohol could be caused to unite with one of dithiocarbimide; the resultant hemithiourethane,  $SCN \cdot CO \cdot NH \cdot CS \cdot OEt$ , was obtained in pale yellow prisms melting at  $141-142^{\circ}$ . It is insoluble in cold water, but decomposes when heated with water to near the boiling-point, carbon dioxide and thiocyanic acid being formed.

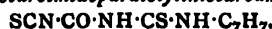
By combination with aniline, compounds having either the formula  $SCN \cdot CO \cdot NH \cdot CS \cdot NHPh$  or  $CO(NH \cdot CS \cdot NHPh)_2$  can be obtained, according to the conditions. The former, carbonylthiocarbimidophenylthiocarbimide, crystallises from alcohol in brilliant, pearly leaflets which melt at  $172^{\circ}$ , then quickly re-solidify, and now melt at  $223^{\circ}$ . It is insoluble in cold water but gradually dissolves on boiling, becoming hydrolysed, carbon dioxide being evolved, whilst thiocyanic acid and phenylthiourea remain dissolved:—



Carbonyldiphenyldithiocarbimide,  $CO(NH \cdot CS \cdot NHPh)_2$ , separates from alcohol in vitreous prisms which melt at  $166^{\circ}$ , and are insoluble in boiling water. Both phenylated derivatives, as well as the hemithiourethane, are readily desulphurised by silver salts and by an alkaline lead solution.

Attempts to produce hemi- or complete thiourethanes by adding carbonyl chloride to potassium or barium thiocyanate in alcoholic solution were unsuccessful; the chloride first interacted with the alcohol, yielding ethyl chlorocarbonate, which, with the metallic thiocyanate, gave carboxyethylthiocarbimide, and this latter, combining with another portion of the alcohol, formed finally Doran's carboxyethylthiourethane (*Trans.*, 1896, lxix., 334) or diethyliminodithiocarbonate,  $EtO \cdot CO \cdot NH \cdot CS \cdot OEt$ . The following compounds were also described:—

Carbonylthiocarbimidoparatolylthiocarbimide,—



—Pearly leaflets melting at  $182^{\circ}$ , re-solidifying at once,

and now melting at 237°. In general properties, this and the naphthyl compound mentioned below closely resemble the phenyl homologue. *Carbonyldi-o-tolylthiocarbamide*,  $\text{CO}(\text{NH}\cdot\text{CS}\cdot\text{NH}\cdot\text{C}_6\text{H}_4)_2$ .—M. p. 172°, has properties similar to those of the corresponding diphenyl compound. *Carbonylthiocarbimido-a-naphthylthiocarbamide*,  $\text{SCN}\cdot\text{CO}\cdot\text{NH}\cdot\text{CS}\cdot\text{NH}\cdot\text{C}_{10}\text{H}_7$ , is a yellowish powder, m. p. 182°, re-solidifying at once and then melting at 222°.

*Carbonylthiocarbimidophenylbenzylthiocarbamide*,—  
 $\text{SCN}\cdot\text{CO}\cdot\text{NH}\cdot\text{CS}\cdot\text{NPhBz}$ .

—Prepared from benzylaniline, it formed long, vitreous prisms having a faint yellow tinge, and melting with effervescence at 180°. When boiled with water it slowly undergoes hydrolysis, carbonic anhydride being evolved, thiocyanic acid passing into solution, and *aa*-phenylbenzylthiourea,  $\text{NH}_2\cdot\text{CS}\cdot\text{NPhBz}$ , being left.

## NOTICES OF BOOKS.

*Logarithmic Tables for Chemists*. ("Logarithmische Rochentafeln für Chemiker"). By Professor Dr. Von F. W. KÜSTER. Dritte neu berechnete und erweiterte Auflage. Leipzig, 1902. Pp. 93.

KÜSTER's logarithmic tables for chemists is a really international scientific work, for the main part is written in a language intelligible to all chemists of the civilised world, in numbers. Even those who do not understand the short explanation to the tables, will see at once that the quantity of lead corresponding to a found quantity of lead sulphate is obtained by adding to the logarithm of the latter the number 83438. But the tables have another international significance, so far as they offer a practical solution of the question which was for the first time discussed in the light of recent work in the *CHEMICAL NEWS* (1889, lviii., p. 307), i.e., what standard should be used in our chemical calculation. It is true that the oxygen standard ( $\text{O}=16$ ) proposed in this journal has been adopted almost universally by chemists, and especially by the International Atomic Weight Commission; but even those chemists who prefer the hydrogen standard can use the present work, as the quotients give absolutely the same practical results, whichever of the two standards is used.

The arrangement of the tables is very practical, and we may not only commend the book for what is contained in it but also for the many superfluities left out. Table I. gives the atomic weights of elements, which are on the whole identical with those of the international table, but containing now and then a decimal more or less, so that the degree of accuracy of the number is seen at once. Tables II. and III. contain the multiple of the more important atomic weights. Tables IV. and V. the weights of the more important molecules, atomic groups, and equivalents and their multiples. Table VI. contains the quotients used for the calculation of the results of analyses, and we are pleased to find that the author has broken with the old and absurd tradition according to which no notice is taken of the "rare elements" like Be, W, Th, &c., or their compounds. In Table VII. are given useful data for measuring gases. Table VIII. refers to the calculation of "indirect" analyses; whilst Table IX. is a modern one, containing several important physico-chemical constants for the calculation of the molecular weight by any of the three methods. Table X. serves for the calculation of the volume of a vessel by "weighing-out" with water or mercury. Table XI. gives the most important electrochemical constants; and Tables XII. and XIII. are devoted to solubilities and to a simple direction for preparing normal solutions from the more concentrated ones. After a few pages of explanatory notes to the tables follow tables of five-figure logarithms, and also another table with four-figures for those who are contented with less

accuracy. Both tables may be trusted, as they are free from misprints. But though a modern chemist may be supposed to know the use of logarithms, as they save his time, and do not so easily lead to error as the old-fashioned methods of multiplication or division, yet the chemist who prefers the older mode of calculation will find the ordinary numbers in the tables prominently printed in red type, whereas the logarithmic values are printed in black, so as to avoid confusion. The reviewer and his students have been using Küster's tables for the last seven years with great advantage in the laboratory, and hopes that others will do the same.

BOHUSLAV BRAUNER.

*The Chemistry of Perfumes and the Manufacture of Essences*. ("Chimie des Parfums et Fabrication des Essences"). By S. PIRASS. With 76 Figures in the Text. Paris: J. B. Baillière et Fils. 1902. Pp. 380.

THIS volume deals with the natural history, chemical composition, and effects of perfumes, and has the advantage of being written by a thoroughly practical man. In this new edition special attention has been paid to artificial perfumes and the preparation of perfumes of known composition.

Perfumes are obtained naturally by expression, distillation, maceration, absorption, and solution; the various processes for carrying out these operations are fully described and illustrated. The third chapter deals with essences, their general characteristic properties, their chemical analysis, and adulteration. For ten years or more chemists have been at work actively in this direction, and a complete account of the facts discovered in this domain will be found in this chapter. The essences are arranged systematically according to the chemical function of the definite compound which plays the principal part in their composition, both from the point of view of their odour and their analysis.

New products are also dealt with, such as synthetic vanilline, artificial musk, ionone, heliotropine, terpineol, &c.

The second part comprises extracts of odours, bouquets, emulsions, pastes, oils, powders, &c.; and finally there is a chapter on substances used in perfumery, such as acetic acid, ammonia, glycerin, vaseline, &c.

*A Text-book of Physics*. By J. H. POYNTING, D.Sc., F.R.S., and J. J. THOMSON, M.A., F.R.S., &c. Properties of Matter. London: Charles Griffin and Co., Ltd. 1902. Pp. 228.

THE volume on "Sound," by the same authors, has been published already, though it is really the second of the series, of which the present volume on the "Properties of Matter" is the first. These will be followed in due course by others on heat, magnetism, and electricity and light.

These text-books are intended for the use of students who lay most stress on the study of the experimental part of physics, though the greater the mathematical knowledge the student possesses, the easier he will find it to follow all the arguments and reasonings to be found in these pages.

This volume is divided into eighteen chapters, and contains 168 illustrations.

The first few chapters deal with weight, mass, and gravitation, and describe a number of historical experiments by means of which the fundamental laws of gravity were proved. Then follow some half-dozen chapters on elasticity, strain, stresses, torsion, spiral-springs, and impact.

Chapter XI. is on the compressibility of liquids, first established by Canton in the year 1762, and Chapters XII. and XIII. deal with the pressure and volume of a gas, and the thermal effects accompanying alterations in strains.

Capillarity is considered in two chapters, Nos. XIV. and XV., and the diffusion of liquids and gases in the two following ones, while Chapter XVIII, and last is on the viscosity of gases.

The volume is of undoubted value and interest, and as would be expected from such well-known authors, the descriptions and reasonings are clear and easily followed, though in some portions of the work the pages of equations have rather a disconcerting appearance. The book is well printed and provided with a good index.

## CORRESPONDENCE.

### FIXING NITROGEN FROM THE ATMOSPHERE.

To the Editor of the Chemical News.

SIR,—In reply to the comments of Mr. Ackroyd (CHEMICAL NEWS, vol. lxxxvi., p. 293) on my note on the above subject, I would mention that my concern was for the oxygen,—"fixing nitrogen" being the title of the original paper. I wrote of an annual 12 million tons of nitrate of soda as a serious additional draft on the oxygen of the atmosphere.

I made the amount 5.65 million tons of oxygen from a total of about 1200 billion tons of oxygen in the atmosphere, which is certainly a very small fraction—and small in comparison with that required by a year's coal production. The amount of coal (British and foreign) raised is stated in "Whitaker's Almanac" to have been nearly 700 million tons in the year 1899. Supposing it to have been equivalent to 70 per cent carbon, and to have been burned to equal volumes of CO and CO<sub>2</sub>, it would require 980 million tons of oxygen. Coal consumption, too, will for a time increase. Much of the oxygen of the nitrate of soda would, no doubt, eventually re-enter the atmosphere as CO<sub>2</sub> (respiration of animal using plant as food, &c.), and its oxygen be partially recovered by vegetation.

The object of my writing on the subject was to remind that, in the interests of posterity, our capital in oxygen, as well as in fuel, may need to be considered.

Lord Kelvin, at the British Association in 1897, in a paper on the fuel supply and air supply of the world, stated (abstract in *Nature*, September 9, 1897, p. 461) that "it followed that the oxygen of the atmosphere resting over Britain is insufficient to burn up the fuel of the country, and the cessation of life may possibly occur by asphyxiation rather than want of fuel."—I am, &c.,

W. H. DERRING.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

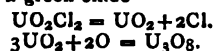
NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxv., No. 21, November 24, 1902.

**Ionisation of a Flame containing Salts.**—Georges Moreau.—The author finds that the unipolar conductivity of a saline vapour is analogous to that of the mass of hydrogen surrounding a filament of incandescent carbon, or that of the gaseous mass which is in contact with a metal illuminated by ultra violet light. In both these latter cases Thomson discovered the production of negative corpuscles on the surface of contact of the metal and of the gas. For saline vapours it seems natural to suppose that these corpuscles are formed at the contact of a negative incandescent electrode. They are detached from the saline molecules probably by the kinetic energy which they

receive from the surface of the metal. A negative charge accelerates their separation; a positive charge retards it. Such corpuscles thrown into the flame ionise the vapour of the salt in the same way as uranium radiations.

**Uranous Oxide.**—Echsnor de Coninck.—If uranyl chloride is calcined in the air it loses its chlorine and is transformed into a green oxide—



The author repeats this experiment with uranyl bromide, and establishes the fact that, even by prolonging the calcination, similar changes do not take place. Uranyl bromide loses all its bromine, and the radicle UO<sub>2</sub> remains in the form of a brick-red mass, which is stable even at high temperatures. When reduced in a current of pure hydrogen this oxide loses traces of water, and is little by little transformed into the black modification. The author therefore concludes—(1) that the uranous oxide when combined with bromine is first transformed into a brick-red modification and then to a black variety; (2) that uranous oxide which exists in uranyl chloride in the state of a radicle is different, in that it is less stable and is transformed into the green oxide by the action of heat. During the calcination of uranyl bromide the evolution of bromine is very exact and allows of an experimental verification of the molecular weight of uranous oxide and the atomic weight of bromine.

**Combinations of Complex Cyanides with the Amines of the Fatty Series.**—P. Chrétien.—By saturating ferrihydrocyanic and ferrihydrocyanic acids with the primary, secondary, and tertiary isoamylamines, a series of well crystalline salts are obtained. Ferrihydrocyanic acid, prepared by the usual method, is employed in aqueous or alcoholic solution; the ferrihydrocyanic acid, prepared by the action of sulphuric acid on the barium salt, in aqueous solution. The author successively adds with 1, 2, 3, and 4 molecules of the amine to one molecule of the first acid, and with 1, 2, or 3 molecules to one molecule of the second, and in each case the product is analysed.

**Estimation of Glycerin in Wine.**—A. Trillat.—The method employed depends on the property which acetic ether possesses, when quite free from all impurities, of dissolving glycerin in the proportion of about 9 per cent at ordinary temperatures, to the exclusion of all the other elements contained in a dry extract of wine.

## MISCELLANEOUS.

**Two Diaries for 1903.**—We have received from the publishers the "Chemists' and Druggists' Diary" and the "Knowledge Diary and Scientific Handbook" for 1903. Besides the usual pages for use simply as a diary, the former contains a great deal of literary matter of interest to chemists and druggists, among which we may mention articles on "Customs Drawbacks," "Excise Licences and Duties," "Formulas for Specialities," "The Medicine Stamp Duty Acts," "Pharmacy and Poison Laws," &c. A number of pages are also devoted to the "Buyer's Guide," which is a complete index to the large number of advertisements in this diary. The "Knowledge Diary" contains, besides a mass of other useful matter, a series of original descriptive articles on various scientific subjects. These include—"Practical Work with the Microscope," by A. Fowler, F.R.A.S.; "Suggestions to Amateur Meteorological Observers," by H. R. Mill, D.Sc.; "Systematic Botany," by R. Lloyd Praeger, B.A.; "Planetary Observation," by W. F. Denning, F.R.A.S.; &c. A very interesting feature of this diary, which we are pleased to see is kept up, is the set of star maps, giving the night sky for one date in every month. There are also maps showing the paths of the planets during the year.

**Wilde Gold Medal and Dalton Medal.**—The Council of the Manchester Literary and Philosophical Society has awarded the Wilde Gold Medal for 1903 to Professor F. W. Clarke, of the United States Geological Survey, and a Dalton Medal to Professor Osborne Reynolds, F.R.S. In special view of the fact that next year will mark the centenary of the discovery by Dalton of the atomic theory, Professor Clarke (whose writings on the atomic weights are so well known) has also been invited, and has consented, to deliver the Wilde Lecture for 1903. The presentation of the medals and the delivery of the lecture will probably take place in May, 1903.

**The Solubility of Salts. Oxalates of Barium.**—E. Groschuff.—Neutral oxalate of barium forms several hydrates. That richest in water is the one represented by  $C_2O_4Ba + 7/2H_2O$ , which occurs in fine needles. The hydrate containing  $2H_2O$  is in hexagonal plates of the clinorhombic system; it generally occurs through the action of pure water on the acid oxalate. The hydrate containing  $1/2H_2O$  is formed at above  $50^\circ$ ; it resembles closely the two others, in clinorhombic prisms. The monohydrate does not appear to exist. The following is an extract from a table of observed solubilities, giving the number of grms. of  $C_2O_4Ba$  dissolved in 1 kilogram. of solution:—

| T.          | Salts at $7/2H_2O$ . | Salts at $2H_2O$ . | Salts at $1/2H_2O$ . |
|-------------|----------------------|--------------------|----------------------|
| $0^\circ$   | .. .. 0.058          | 0.053              | 0.087                |
| $18^\circ$  | .. .. 0.112          | 0.089              | 0.124                |
| $30^\circ$  | .. .. 0.170          | 0.121              | 0.140                |
| $50^\circ$  | .. .. —              | —                  | 0.164                |
| $100^\circ$ | .. .. —              | —                  | 0.211                |

As for the acid oxalate,  $C_2O_4Ba.C_2O_4H_2 + 2H_2O$  or  $+ H_2O$ , we cannot say for certain that it has a known solubility in water, as it is decomposed by the latter into oxalic acid and the neutral salt, and the reaction is reversible. For example, we get—

| T.         | Grms. of acid salt decomposed by 100 grms. of water. | Grms. of water necessary to decompose 1 grm. of acid salt. |
|------------|------------------------------------------------------|------------------------------------------------------------|
| $0^\circ$  | .. .. 1.06                                           | 94.7                                                       |
| $18^\circ$ | .. .. 2.59                                           | 38.6                                                       |
| $60^\circ$ | .. .. 14.6                                           | 6.85                                                       |
| $99^\circ$ | .. .. 46.6                                           | 2.15                                                       |

—*Berichte*, vol. xxxiv., p. 3313.

**Oxycelluloses.**—A. Nastukoff.—Under the name of  $\beta$ -oxycelluloses, we include those oxycelluloses soluble in ammonia and obtained by the action of nitric acid of 1.3 density on cellulose or on cellulosic materials. By using 2.5 parts of nitric acid, the author obtained a return of 90 per cent; this return diminishes if we increase the amount of acid used, and oxalic acid is then formed. The  $\beta$ -oxycelluloses give a barium salt containing about 5 per cent of barium; this gives us an idea of the molecular size of the  $\beta$ -oxycelluloses, and at the same time enables us to distinguish them from the  $\gamma$ -oxycelluloses, of which the salts of barium contain about 1 per cent of the metal.—*Berichte*, vol. xxxiv., p. 3589.

**Autoxidation of Non-saturated Hydrocarbides.**—C. Engler and W. Frankenstein.—The fulvenic carbides are particularly susceptible to spontaneous oxidation in the air. Thus, when we make a 5 to 7 per cent solution of dimethylfulvene,  $(CH_3)_2C=C_5H_4$ , in benzene, and shake it up thoroughly in a flask for several days in contact with air, we observe that the liquid becomes cloudy and then deposits a granular white precipitate; the reaction is helped by light or by a gentle heat. The product formed is a diperoxide with the formula  $C_8H_{10}O_4$ ; it is a white powder, insoluble in most of the organic solvents, and detonating if heated to  $130^\circ$  or with concentrated sulphuric acid, besides being spontaneously decomposable. The same takes place with methylethylfulvene (an orange liquid boiling at  $185^\circ$ ); a similar peroxide is formed, but the reaction is less rapid. No definite product of reaction

is produced with cyclopentadiene, nor with hexylene, &c. Experiments in oxidation with all the different carbides in the presence of indigo-carmin, have shown that indigo fixes a quantity of oxygen equal to half that which becomes joined to the carbide.—*Berichte*, vol. xxxiv., p. 2933.

## NOTES AND QUERIES.

\*. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

**Potassium Persulphate.**—(Reply to "S. C.")—"S. C." asks for a method of preparing potassium persulphate; also for probable cause of failure of his own apparatus. The condition necessary for a large and quick yield is a high current density at the anode. The very small current density obtained by using a platinum dish as anode explains the failure of "S. C.'s" apparatus. The writer used for cathode a sheet of platinum-foil rolled into a cylinder inside a cylindrical porous earthenware vessel filled with dilute  $H_2SO_4$ . This was placed in a glass jar containing a saturated solution of  $KHSO_4$ , in which the anode, a platinum rod, was hung. The whole apparatus was placed in a tin vessel, through which cold water was circulated, and the inner porous cell was cooled by a worm of glass tubing through which cold water ran. A current of 4.5 amperes was used; the temperature being about  $12^\circ C$ . The persulphate, formed in three to four hours, was filtered off and re-crystallised from hot water; yield, 60 to 70 per cent. With ammonium sulphate the precipitate comes in from fifteen to twenty minutes.—CHAS. S. PATTERSON, Chemistry Building, McGill University, Montreal, November 25, 1902.

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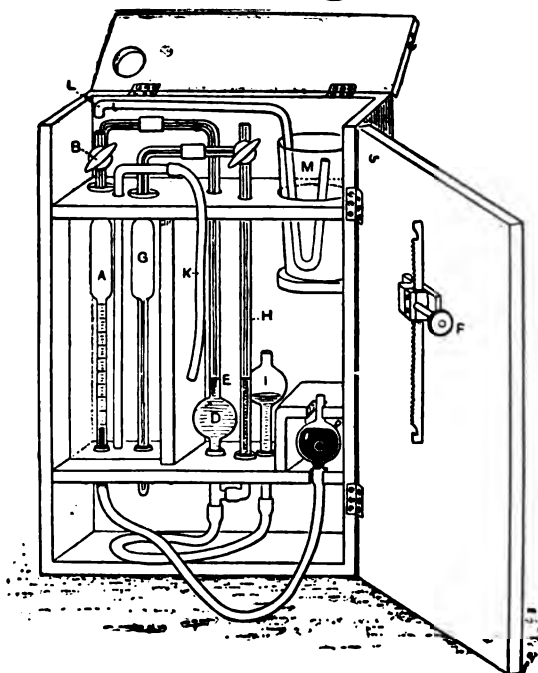
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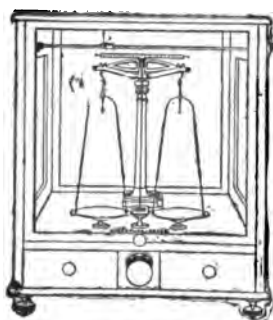
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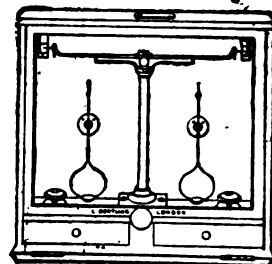


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# THE CHEMICAL NEWS

VOL. LXXXVI., No. 2248.

## NOTE OF THE COMPOSITION OF A JERSEY SOIL.

By WILLIAM NAYLOR, F.C.S., A.M.I.C.E.

THE following notes on the chemical composition of a soil from Millbrook, Jersey, Channel Islands, may be worth recording for the use of agricultural chemists who may be called upon in the future to examine soils in this neighbourhood.

The examination was made at the instance of the estate owner, who had reason to believe that it did not respond properly to the variations in the application of nitrogenous and phosphatic manures. This well-defined object of examination, as well as the plainly evident physical character of it—a loamy soil from over a shale formation—rendered any special examination of the moisture absorbing or retentive power, evaporative efficiency, heat absorbing or radiating capacity unnecessary. The sample was drawn this year after the gathering of the potato crop; the particulars of which crop, as well as the manuring, will be given later.

The mechanical examination, after drying four or five days at 80°–90° C., gave the following:—

|                                             | Per cent. |
|---------------------------------------------|-----------|
| Coarse gravel, retained by 1/10 inch mesh.. | 2'322     |
| Consisting of—                              |           |
| Mineral matter (ammonio-carbonated) ..      | 2'084     |
| Moisture and organic loss (ignition) ..     | 0'238     |
| Fine gravel, retained by 1/50 inch mesh ..  | 6'544     |
| Consisting of—                              |           |
| Mineral .. .. .                             | 5'896     |
| Organic .. .. .                             | 0'648     |
| Fine sand and clay, passed by 1/50 in. mesh | 91'134    |
| Consisting of—                              |           |
| Mineral .. .. .                             | 83'720    |
| Organic .. .. .                             | 7'414     |
| Specific gravity .. .. .                    | 2'441     |

The slightly higher proportion of loss in the coarser material would be probably due to moisture in the "lumps," for when a representative sample was taken of the air-dried, powdered to pass through a sieve with 2500 holes to the square inch and further dried, the following figures were obtained:—

|                        | Per cent. |
|------------------------|-----------|
| Moisture .. .. .       | 1'800     |
| Organic matter .. .. . | 6'330     |
| Mineral matter .. .. . | 91'870    |

For determining the solubility of the mineral residue in acid three separate portions were digested: the first for one day, another during two days, and the last during three days. Hydrochloric acid was used of sp. gr. 1'115.

The differences between each day's figures were but slight after the first day (it was found up to six or seven days); the second of the following three being accepted:—

### Matters Soluble in Acid, given as Percentages of the Soil.

|                                 | One day. | Two days. | Three days. |
|---------------------------------|----------|-----------|-------------|
| Combined silica .. ..           | 0'011    | 0'012     | 0'010       |
| Soda (Na <sub>2</sub> O) .. ..  | 0'101    | 0'137     | 0'136       |
| Magnesia (MgO) .. ..            | 0'206    | 0'180     | 0'180       |
| Lime (CaO) .. ..                | 0'300    | 0'350     | 0'350       |
| Potash (K <sub>2</sub> O) .. .. | 0'385    | 0'340     | 0'288       |
| Iron and alumina .. ..          | 3'700    | 3'850     | 3'500       |
| Phosphoric anhydride ..         | 0'206    | 0'206     | 0'206       |
| Sulphuric anhydride ..          | 0'154    | 0'140     | 0'171       |
| Leaving matters insoluble .. .. | 87'030   | 86'600    | 86'800      |

The small quantities of alkalis and alkaline earths present (the whole amounting to only 1 per cent in a humus soil, well manured), is striking, and led to an examination of the soil-producing constituents—the matters insoluble in acids. These, after fusing with alkaline carbonates (barium carbonate separately for the potash estimation), gave the following:—

### Matters Insoluble in Acid, as Percentages of the Soil.

|                            |        |
|----------------------------|--------|
| Soda and potash .. .. .    | 0'000  |
| Magnesia (MgO) .. .. .     | 0'250  |
| Lime (CaO) .. .. .         | 0'496  |
| Iron and alumina .. .. .   | 13'142 |
| Sulphuric anhydride .. ..  | 0'432  |
| Phosphoric anhydride .. .. | 0'000  |
| Silica .. .. .             | 69'905 |

The proportion of lime and magnesia in the soluble as compared with the insoluble is therefore less, in spite of manuring with superphosphate to the extent of 10 cwts. per acre. Although the insoluble portion evidently affords some small reserve fund of lime and magnesia from which the soil may draw to a slight extent, it is very small, and a sample of the rock formation (shale) was then examined with the object of discovering whether this might afford more. This examination yielded the following figures:—

|                                   |        |
|-----------------------------------|--------|
| Soluble in acid—                  |        |
| Soda (Na <sub>2</sub> O) .. .. .  | 0'239  |
| Potash (K <sub>2</sub> O) .. .. . | 0'346  |
| Lime (CaO) .. .. .                | 0'450  |
| Magnesia (MgO) .. .. .            | 0'936  |
| Iron and alumina .. .. .          | 9'700  |
| Sulphuric anhydride .. ..         | 0'068  |
| Phosphoric anhydride .. ..        | 0'095  |
| Loss on ignition .. .. .          | 4'100  |
| Insoluble in acid .. .. .         | 83'450 |

Of the rock formation, the matter insoluble in acid contained—

|                            |        |
|----------------------------|--------|
| Magnesia .. .. .           | 0'271  |
| Lime .. .. .               | 0'466  |
| Soda and potash .. .. .    | —      |
| Phosphoric anhydride .. .. | —      |
| Sulphuric anhydride .. ..  | 0'610  |
| Iron and alumina .. .. .   | 25'779 |
| Silica .. .. .             | 51'219 |

It appears clear, then, that the rock formation, in the slow process of disintegration, is likely to afford some little more of the alkaline earths, but not much; and, comparing the tables, it will be noticed that the impoverishment of importance is in respect to lime, magnesia, and soda. By means of the manures used, the potash natural *quantum* is about maintained, while the phosphoric and sulphuric acid figures are augmented.

Although it is well known that, in many cases, it is economical to add more manure than is actually required for plant nutrition, owing to its beneficial action with regard to the moisture-retaining qualities of the soil, its invigorating action on the rootlets or the like, and that any calculation of the actual manure required for absorption based on the ash constituent of the crop is misleading, it may be logical to calculate the *proportions* of nutrient material added and compare them with the ash constituent of the crop, especially where a certain ash constituent of the soil is growing beautifully less.

The following was the manuring of the land in question:—

|                                                        |                                         |
|--------------------------------------------------------|-----------------------------------------|
| 10 cwt. bone-meal (superphosphate), equal to .. ..     | 222½ lbs. P <sub>2</sub> O <sub>5</sub> |
| 5 cwt. sulphate of potash, 95 per cent, equal to .. .. | 160 lbs. of lime                        |
| 7½ cwt. sulphate of ammonia, equal to .. ..            | 287 lbs. K <sub>2</sub> O               |
|                                                        | 203 lbs. sulphur                        |
|                                                        | 203 lbs. sulphur                        |
|                                                        | 179 lbs. nitrogen                       |

306

The crop was 12,000 lbs. of potatoes, which pulled very young would have quite doubled if not more than the usual quantity of haum per given weight of tuber. The ash content, then, as compared with the manure added, would be as follows:—

|                         | Ash,<br>lbs. per acre. | Manure applied,<br>lbs. per acre. |
|-------------------------|------------------------|-----------------------------------|
| Potash .. .. .          | 76                     | 287                               |
| Lime .. .. .            | 48                     | 160                               |
| Phosphoric anhydride .. | 25                     | 222½                              |
| Magnesia .. .. .        | 31                     | —                                 |
| Soda .. .. .            | 7½                     | —                                 |
| Sulphur .. .. .         | 6½                     | 306                               |
| Nitrogen .. .. .        | 85                     | 179                               |

(Farmyard manure also added copiously).

Magnesia and lime therefore show up badly—in fact, worst—and in the presence of so much sulphuric acid, in a form not the most easily decomposed. The wash-water from the soil was neutral to litmus. The soluble portion from this sample of soil is compared in the following table with one from a neighbouring estate not so freely manured, but giving better crops—taken after the pulling of the crop. I have reason to believe that, in the latter case, a dressing of chalk is given periodically.

#### Portion Soluble in Acid.

|                         | Soil No. 1. | Neighbouring estate. |
|-------------------------|-------------|----------------------|
| Organic matter .. ..    | 6'330       | 4'800                |
| Organic nitrogen .. ..  | 0'154       | 0'112                |
| Sulphuric anhydride ..  | 0'208       | 0'000                |
| Phosphoric anhydride .. | 0'140       | 0'066                |
| Potash .. .. .          | 0'340       | 0'231                |
| Soda .. .. .            | 0'137       | 0'071                |
| Lime .. .. .            | 0'350       | 0'500                |
| Magnesia .. .. .        | 0'180       | 0'180                |

### ON CERTAIN TELLURIUM MINERALS, AND THE ACTION OF SULPHUR MONOCHLORIDE THEREON.

By R. W. EMERSON MACIVOR, F.I.C., F.G.C.S., &c.,  
Formerly Assistant Professor of Chemistry, Anderson's College  
(Medical Faculty), Glasgow.

SULPHUR monochloride readily acts on tellurium with evolution of heat, and according to the proportions in which the two substances are brought together, there results either  $\text{TeCl}$  or  $\text{TeCl}_4$ , and of course free sulphur. Kraft and Steiner, who first studied the reaction (*Berichte der Deutsch. Chem. Ges.*, xxiv., 560), by heating two parts  $\text{S}_2\text{Cl}_2$  with 2.2 parts of tellurium, obtained the black amorphous  $\text{TeCl}_2\text{S}_2\text{Cl}_2 + \text{Te} = \text{TeCl}_2 + 2\text{S}$ . In this experiment the proportion of Te employed was in excess of that actually required by the equation. But Lenher has shown (*Journ. Am. Chem. Soc.*, xxiv., 189) that, provided an excess of  $\text{S}_2\text{Cl}_2$  is employed, the Te is converted into the highly crystalline tetrachloride,  $2\text{S}_2\text{Cl}_2 + \text{Te} = \text{TeCl}_4 + 4\text{S}$ . This reaction takes place at any temperature from the ordinary boiling-point of  $\text{S}_2\text{Cl}_2$ , viz.,  $139^\circ\text{C}$ .; the sulphur produced dissolving in the excess of  $\text{S}_2\text{Cl}_2$ , while the  $\text{TeCl}_4$ —which is practically not soluble in the liquid in the cold, though readily so in the heat—separates out on cooling in needle-shaped crystals. Lenher states that the yield of  $\text{TeCl}_4$  by this reaction is in nature almost quantitative, and my own results will be seen to quite confirm this opinion.

It occurred to me that it might be not altogether uninteresting to try the action of  $\text{S}_2\text{Cl}_2$  on the specimen of native tellurium described by me in the columns of the CHEMICAL NEWS some time ago (vol. lxxxii., p. 272), and also on a remarkably pure sample of calaverite from Kalgoorlie, East Coolgardie, W. Australia, kindly sent me two years ago by Mr. Henry Pittman, B.Sc.

The native tellurium had a specific gravity = 6.22, and contained 96.94 per cent of Te and 2.4 per cent of gold. It was first reduced to the finest possible powder, and 2.85 grms. were heated in a large excess of  $\text{S}_2\text{Cl}_2$  in an open tube for about an hour, when the whole of the Te was found to have been transformed into tetrachloride, and on cooling crystallised out completely. The excess of  $\text{S}_2\text{Cl}_2$ , containing the dissolved sulphur, was carefully drained off, and the crystals of  $\text{TeCl}_4$  thoroughly washed with  $\text{CS}_2$ —in which the substance is insoluble—to remove all traces of  $\text{S}_2\text{Cl}_2$  and S. The tetrachloride obtained had a yellow-grey colour, owing to the impurity in the original mineral—but on volatilisation in a current of pure dry  $\text{CO}_2$  condensed in beautiful white needles melting at  $214.5^\circ$ . On testing the small residue left behind in the tube after the complete vaporisation of the  $\text{TeCl}_4$  it was found to contain only a mere trace of tellurium.

The calaverite used in these experiments had a specific gravity = 9.314, and consisted of:—

|            |       |
|------------|-------|
| Te .. .. . | 57.00 |
| Au .. .. . | 42.15 |
| Ag .. .. . | 0.60  |
|            | 99.84 |

Some 4 grms. of the finely-ground mineral were digested in excess of  $\text{S}_2\text{Cl}_2$  at about  $100^\circ$ , and only after prolonged heating did I succeed in effecting a complete decomposition. The  $\text{TeCl}_4$  separated on cooling, and was freed from the  $\text{S}_2\text{Cl}_2$  and S by decantation and thorough washing with  $\text{CS}_2$ . The tetrachloride was dissolved in  $\text{HCl}$ , the solution filtered, and the Te thrown down by aid of acid sulphite of sodium. The precipitate was collected by reversed filtration on a tared filter, dried at  $120^\circ\text{C}$ ., and weighed. I recovered 2.15 grms. of Te, equal to 94.3 per cent of the total amount originally present in the calaverite—a fairly satisfactory result when the circumstances under which I worked are taken into consideration.

The residue left in the tube and on the filter after the extraction of the  $\text{TeCl}_4$  by  $\text{HCl}$  was examined, and found to contain a small amount of tellurium, possibly held in combination by the silver present.

In the course of these experiments I had occasion to make some trials of the various processes for estimating tellurium in the elementary condition, but the results obtained I intend to make the subject of a separate paper.

The Laboratory, 29, Fenchurch Street, E.C.

**Sorbic Acid.**—O. Dœbner and A. Voif. —Sorbic acid has been prepared by the method already described (*Berichte*, vol. xxiii., p. 2140) from malonic acid, crotonic aldehyde, and pyridine. **Methylic Ether.**—Prepared from sorbic chloride and methylic alcohol; it is a liquid with an odour of aniseed, boiling at  $174^\circ$  at the ordinary pressure, and at  $70^\circ$  under 20 m.m.; it solidifies in a freezing mixture, and fuses at  $+5^\circ$ . **Chloride of Sorbyl.**—One part of sorbic acid and two parts of  $\text{PCl}_5$  are rubbed up together in a porcelain mortar; the resulting liquid is fractionated under 20 m.m.; chloride of sorbyl boils at  $78^\circ$  under 15 m.m.; the return is from 80 to 85 per cent of the weight of acid used. **Sorbamides.**—Chloride of sorbyl is added drop by drop to aqueous ammonia, concentrated, and cooled with ice. It forms fine needles from a boiling solution in water, or hard prisms from alcohol, fusible at  $168^\circ$ . **Sorbaniilide.**—Formed from sorbyl and aniline, fusible at  $153^\circ$ . **Sorbonitrile.**—Two parts of sorbamide and one part of  $\text{P}_2\text{O}_5$  are heated in an oil-bath at  $150^\circ$ ; it is fractionated *in vacuo*; it occurs as a colourless oil, boiling at  $72^\circ$  under 20 m.m. **Ketone.**  $\text{C}_8\text{H}_5\text{—CO—CH=CH—CH=CH—CH}_3$ .—Prepared by means of chloride of sorbyl and zinc-ethyl; the product is decomposed by water, and carried off by steam. The ketone is separated from the distillate by the addition of sea-salt; it is a clear, yellow oil, boiling at  $90\text{—}95^\circ$  under 26 m.m.—*Berichte*, vol. xxxiv., p. 2221.

# ACTION OF TELLURIUM AND SELENIUM ON GOLD AND SILVER SALTS.

By R. D. HALL and VICTOR LENHER.

## Historical.

THAT metallic tellurium and selenium act as reducing agents upon solutions of gold and silver salts has been known since the early part of the last century, still their action has not been studied quantitatively.

N. W. Fischer (*Pogg. Ann.*, xii., 502) found that tellurium acts as a reducing agent on salts of gold, silver, platinum, and palladium in solution, the reduction being incomplete in all cases, and that its action on gold solutions is the most rapid, the tellurium after a short time becoming coated with metallic gold, stopping all further action even at a high temperature. Fischer states that the action of tellurium on silver nitrate in solution is slower than on a gold solution, and that the black powder which remains is not silver but a union of tellurium and silver, each in the lowest state of oxidation. He also observes that selenium reduces a gold solution at a high temperature, the action then being the same as that of tellurium, while silver and the remaining metals are not reduced.

Parkham (*Chem. Centrbl.*, xxxiii., 813) found that red selenium is blackened in a solution of silver nitrate, some flakes of selenious acid being formed at the same time; after the latter has been removed by sodium hydroxide the black powder which remains contains both selenium and silver in quantity, but no unchanged selenium is visible under the microscope. He notes that tellurium in a silver nitrate solution gives a black precipitate which shows no metallic lustre under pressure. After digesting tellurium for four days with an excess of a saturated silver nitrate solution he obtained a precipitate, which appeared homogeneous under the microscope, contained both tellurium and silver and no tellurous acid, and concludes that if metallic silver is precipitated by tellurium, then under these conditions nearly all of the tellurium should be in solution. He analysed neither of the precipitates, but he found that selenium in a copper solution gives a precipitate which analysed for  $\text{Cu}_2\text{Se}$ , and that tellurium in a copper solution gives, according to conditions,  $\text{Cu}_2\text{Te}$  or  $\text{CuTe}$ , and because of this he concludes that selenium in silver solutions forms silver selenide,  $\text{Ag}_2\text{Se}$ , and that tellurium in silver solutions precipitates  $\text{Ag}_2\text{Te}$ , the telluride of silver.

Senderens (*Comptes Rendus*, civ., 175) found that selenium reduces a boiling solution of silver nitrate, either dilute or concentrated, with the formation of silver selenide and selenium dioxide. When the reaction was carried out in a closed tube, he obtained a blue colour which he thought indicated the formation of nitrogen pentoxide. The action of tellurium on a silver nitrate solution was found to be less rapid than that of selenium at  $100^\circ$  but similar to it; also at ordinary temperatures both metals reduce silver nitrate, the action being slow but complete.

## Procedure.

The following investigation was undertaken to find out whether gold and silver are precipitated from solutions of their salts by elementary tellurium and selenium. Pure tellurium and selenium were used. They were ground to a powder in an agate mortar and weighed quantities, usually about 0.1 gm., of them kept in contact with the solution of gold or silver for a definite length of time under definite conditions. The precipitate obtained was collected on a Gooch filter, dried at  $100^\circ$  in an air-bath, and weighed; the precipitate was usually tested for either metal that it might contain, and in some cases both the precipitate and the solution were analysed. In most cases solutions containing known quantities of silver and gold were used, so that all the data necessary for an analysis was the determination of the amount of these metals remaining in the solution.

## Tellurium in Gold Solutions.

In preliminary experiments on the reducing action of tellurium on solutions of gold chloride, considerable difficulty was first experienced in obtaining results consistent with the equation  $3\text{Te} + 4\text{AuCl}_3 = 3\text{TeCl}_4 + 4\text{Au}$ , since when tellurium is brought in contact with a gold solution the gold is apt to deposit as a coating on the tellurium, protecting it from all further action, a fact also noted by Fischer. This was in part obviated by grinding the tellurium to an impalpable powder, and even when this was done the tellurium was apt to mass in little balls which it was necessary to crush with a glass rod in order to bring all of the tellurium in contact with the gold solution. That some tellurium not infrequently escaped contact with the gold solution was shown by extracting the precipitate with such solvents as sulphur monochloride and nitric acid. None could be dissolved, showing that the tellurium present was completely enclosed by the gold; but when the precipitate was dissolved in aqua regia, the nitric acid evaporated off and the tellurium and gold precipitated with sulphur dioxide, and this precipitate either tested for tellurium by the above solvents or by fusion with potassium nitrate, invariably where the weight of the precipitate was below that required by the above equation, tellurium could be detected.

It is not necessary that the gold chloride solution should contain more gold than is required by the reaction, for if a large excess of gold solution is used the results are the same, while if an insufficient supply is used it is entirely bleached, all of the gold being deposited. The action of tellurium on gold in solution is fairly rapid; warming a few minutes will bring down sufficient gold to colour the precipitate a decided yellow, yet time is a considerable factor in bringing about complete precipitation, from two to three hours being necessary with continued heating, or several days at room temperatures. The only conditions to be observed, in order to obtain quantitative precipitation of the gold by the tellurium, are sufficient time and the direct contact of all the tellurium with the gold solution.

**Experiment 1.**—0.1010 gm. of tellurium gave 0.2125 gm. of gold; the theory, for total precipitation of the gold by the tellurium according to the equation given, requires 0.2091 gm. The gold solution used contained 0.4346 gm. of gold and was in contact with the tellurium for three hours and was boiled continuously during that time. No test for tellurium could be obtained in the precipitate either before or after dissolving in aqua regia.

**Experiment 2.**—0.1099 gm. of tellurium gave 0.2257 gm. of precipitate; the theory requires 0.2243 gm. The gold solution contained 6.2351 gm. of gold and was in contact with the tellurium for six days at room temperature. The precipitate contained no tellurium.

**Experiment 3.**—0.0789 gm. of tellurium gave 0.1642 gm. of gold; the theory requires 0.1635 gm. The precipitate gave no test for tellurium either before or after dissolving in aqua regia.

## Selenium in Gold Solutions.

The selenium used was prepared from sublimed selenium dioxide by precipitation with sulphur dioxide in the presence of hydrochloric acid. The selenium was dried, fused, and ground to a fine powder. The fused variety of selenium does not reduce a solution of gold chloride at room temperature. One experiment was made allowing the selenium to remain in contact with the gold solution for three months at room temperature with no visible action. At the boiling temperature the action is nearly as energetic as that of tellurium and is analogous to it, being expressed by the reaction—



The same difficulties were encountered with the selenium as with the tellurium and were overcome in the same way. From six to eight hours of continued boiling are necessary

to insure complete precipitation, or better from two to three days at a temperature of from 70° to 80°.

*Experiment 1.*—0.0712 grm. of selenium gave 0.2345 grm. of gold, the calculated being 0.2336 grm.

*Experiment 2.*—0.1058 grm. of selenium gave 0.3455 grm. of precipitate; the theory requires 0.3470 grm. The amount of gold contained in the solution was 0.4346 grm. and it was in contact with the selenium for three days at a temperature of about 80°. The precipitate gave no test for selenium after dissolving and re-precipitating.

*Experiment 3.*—0.1502 grm. of selenium in a solution of gold chloride boiled for five hours gave 0.4892 grm. of gold; the amount required by the theory is 0.4926 grm.; the precipitate gave no test for selenium.

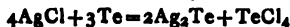
#### *Tellurium in Silver Solutions.*

The first salts used were ammoniacal solutions of the nitrate and the chloride of silver. In both cases the solutions were reduced by the tellurium, slowly in the cold and more rapidly if heated, yet tellurium digested with silver nitrate solution for a long time would not completely go into solution leaving metallic silver. Where an ammoniacal solution of silver nitrate was used the weight of the precipitate and the amount of silver in the precipitate was in excess of that required for silver telluride. The amount of silver and tellurium as determined by analysis did not add up to the total weight of the precipitate, and in several cases small plates of metallic silver were formed. On repeating the experiment with no tellurium present it was found that, when silver nitrate stands in contact with ammonia for some time or is warmed with it, a deposit is formed which is insoluble in ammonia. This deposit is probably silver nitride, but when the solution is boiled or kept warm several days the precipitate contains plates of metallic silver. In subsequent experiments the silver nitrate solution was used without ammonia, and the silver tellurite formed was removed by washing the precipitate with ammonia.

When silver chloride in ammonia was used, it was allowed to stand in contact with the tellurium at room temperature or only slightly warmed, an excess of ammonia always being present to keep the silver chloride in solution. When the nitrate was used a greater range of temperature was possible, and the solution was either boiled continuously or kept at a temperature of about 80° for a long time. At the end of the experiment the precipitate was collected on a Gooch filter, washed several times with ammonia to remove any silver tellurite, and then with water. In this way results were obtained which agree very well with those required by the equation—



where the nitrate of silver is used, and—



where the chloride in ammonia was used. Either the chloride or the nitrate of tellurium would be decomposed by water and would then react with more silver nitrate or chloride to form the tellurite of silver.

*Experiment 1.*—0.1099 grm. of tellurium gave 0.1848 grm. of precipitate; the calculated amount of silver telluride was 0.1978 grm. The solution contained 0.4320 grm. of silver as the chloride and was in contact with the tellurium for eight days.

*Experiment 2.*—0.1005 grm. of tellurium in a solution of silver chloride containing 0.2160 grm. of silver gave 0.1843 grm. of precipitate, that required for silver telluride being 0.1809 grm.; the time of the experiment was ten days.

*Experiment 3.*—0.0994 grm. of tellurium in a solution of silver chloride containing 1.0800 grm. of silver gave 0.1767 grm. of precipitate, the calculated for silver telluride being 0.1789 grm. The time of the experiment was three months, and during that time it was kept in a warm place and ammonia added occasionally.

*Experiment 4.*—0.1018 grm. of tellurium in a solution of silver nitrate which contained 2.0601 grm. of silver gave 0.1871 grm. of precipitate, that required for silver telluride

being 0.1841 grm. The silver remaining in the solution was determined as the chloride and found to be 1.9404 grm., which would make the loss from the solution or the amount of silver in the precipitate as 0.1192 grm. and the amount of tellurium in the precipitate 0.0693 grm. The calculated amount of silver in 0.1871 grm. of the telluride is 0.1178 grm. and the tellurium 0.0693 grm.

The low results in the above series of experiments can be attributed in a large measure to small amounts of tellurium which were so thoroughly enclosed in the mass that they could not be brought to enter into the reaction.

An attempt was made to prepare silver telluride in order to study its properties and compare it with the precipitate obtained from the reduction of the silver solution by tellurium. An excess of aluminum was fused with tellurium and hydrogen telluride evolved from this by treatment with dilute hydrochloric acid. The hydrogen telluride was passed into an ammoniacal solution of silver nitrate in an atmosphere of carbon dioxide to prevent any decomposition of the hydrogen telluride by the oxygen of the air. This gave a black precipitate which was washed repeatedly with water, then dried in an air-bath at 105°. Analysis showed the substance to contain 22.3 per cent tellurium and 77.3 per cent of silver, while the theory for silver telluride requires 37.1 per cent of tellurium and 62.9 per cent of silver. The silver in another sample, prepared in a similar way, was determined by heating the substance in a boat in a current of chlorine gas; the tellurium distilling off as the tetrachloride and the silver chloride remaining was weighed. The per cent of silver obtained in this way was 75.4.

The substance thus prepared fused without loss of tellurium, as was indicated by the absence of fumes, to a porous mass which cut with a metallic lustre. It was acted on by sulphur monochloride, forming tellurium tetrachloride; it reduced a gold solution, depositing metallic gold, but no action could be observed with a silver solution.

The action of hydrogen telluride on silver nitrate may be explained by the decomposition of hydrogen telluride in presence of silver nitrate into nascent hydrogen and tellurium, and these then would reduce the silver solution, the first causing metallic silver to be deposited while the latter would give rise to silver telluride, and this would explain the high percentage of silver.

Silver tellurite was prepared by adding potassium tellurite (obtained by fusion of equivalent weights of tellurium dioxide and potassium carbonate) to a silver nitrate solution; it was dried at 105° and as obtained was a light yellow powder, fairly permanent in the light. Analysis gave 54.95 per cent of silver, while the formula  $\text{Ag}_2\text{TeO}_3$  require 55.17 per cent. This silver tellurite was reduced by heating it in a porcelain tube to the highest temperature obtainable in the blast-lamp and passing a current of dry ammonia over it for one hour. At the temperature used, tellurium alone would have entirely distilled from the boat. 1.1990 grms. of the silver tellurite were used and 1.0440 grms. of residue obtained; the weight required for silver telluride from the amount of telluride used is 1.0517 grms. The compound obtained by the reduction of the tellurite was analysed by heating in a current of chlorine. 0.2276 grm. gave 0.1908 grm. of silver chloride or 62.65 per cent of silver, the calculated for silver telluride being 62.97 per cent. Another sample of silver tellurite was reduced in carbon monoxide under similar conditions, and the telluride formed was similar in all respects to that obtained by reduction in ammonia.

The silver telluride obtained by the reduction of silver tellurite had a strong metallic lustre, but did not tarnish readily, was harder than tellurium, was brittle not brittle enough to be ground to a powder; it was broken up as finely as possible and treated with sulphur monochloride, gold chloride solution, and silver nitrate solution. Sulphur monochloride acted on it slowly to form tetrachloride; the gold solution was bleached and the silver telluride became coated with gold. 0.2995 grm. was

boiled with a solution of silver nitrate for eight days; at the end of that time the solution gave no test for tellurium, while the particles had lost none of their lustre, showing them to be entirely unacted on.

Brauner (*Journ. Chem. Soc.*, lv., 388) synthesised silver telluride by fusion of silver in a current of tellurium. He obtained a crystalline mass of metallic lustre having approximately the composition  $\text{Ag}_2\text{Te}$ .

In an Article on the "Naturally Occurring Telluride of Gold" by one of us (*Journ. Am. Chem. Soc.*, xxiv., 355) specimens of sylvanite, calaverite, coloradoite, kalgoorlite, and nagyagite were treated with a gold solution, and in all cases metallic gold was obtained. Recently some excellent specimens of krennerite, hessite, and more specimens of sylvanite and calaverite were obtained, and as specimens of some of these minerals were not at hand at the time of the previous investigation their action on solutions of gold chloride and silver nitrate was now tried. The minerals examined were:—

One specimen calaverite, Keystone, Boulder Co., Col. (this was one of the original specimens of Genth).

One specimen calaverite and one of sylvanite from Cripple Creek, Col.

Three specimens hessite from Boles, Siebenburgen, Hungary.

One specimen hessite from Tombstone, Arizona.

One specimen krennerite, West Side Mine, Cripple Creek, Col.

One specimen krennerite, Boles, Sidenburgen, Hungary.

All of these minerals reduced gold chloride solution, becoming coated yellow, and where a small quantity of solution was used bleached it, agreeing with the previous results. The hessite was without visible action on the silver nitrate solution even when boiled in it; one specimen of krennerite and one of sylvanite gave a small amount of black powder similar to that obtained from tellurium in silver nitrate solution. Hessite is telluride of silver, which as we have seen is without action on silver solutions, while the others contain both silver and gold with the tellurium, or gold with tellurium, from which it would seem that, while the combination of silver with tellurium is without action on silver nitrate solution, that of gold with tellurium reduces silver nitrate.

In the same article reference was made to the action of tellurium on silver nitrate solution, and conclusions stated which in the light of the facts then at hand seemed correct. It was stated that the action of tellurium on silver solutions is similar to that on gold solutions, though not as complete, metallic silver being formed. The results in this later work show that tellurium in silver solutions forms the telluride of silver and not metallic silver. The error in the former statements arose from the use of ammonia in the solution while it was in contact with the tellurium; it was subsequently found that silver nitrate solution standing for a long time with ammonia, or being heated with it, gives a deposit which contains metallic silver.

#### Selenium in Silver Solutions.

Selenium reduces a solution of silver nitrate or of the chloride in ammonia, either in the cold or on heating. The action is less rapid than that of tellurium but similar to it, a close approximation to the selenide of silver being formed. Parkham's statement that a precipitate of selenium dioxide is formed is obviously incorrect, as he used aqueous solutions, and selenium dioxide is readily soluble in water. The precipitate that he obtained must have been silver selenite. Senderens' experiment of heating the solution in contact with the selenium in a closed tube was repeated; no difference could be detected in the outcome of the reaction, and no blue colouration was observed.

Experiment 1.—0.1012 grm. of selenium in a solution of silver nitrate for eight days gave 0.2513 grm. of precipitate, the theory requiring 0.2497 grm.

Experiment 2.—0.1047 grm. of selenium in a solution of silver chloride containing 0.4320 grm. of silver, gave 0.2550 grm. of precipitate, the calculated for silver selenide being 0.2590 grm.

Experiment 3.—0.1012 grm. of selenium in a solution of silver nitrate containing 2.4157 grms. of silver gave 0.2468 grm. of precipitate, the theory for silver selenide being 0.2497 grm. After the removal of the precipitate the solution contained 2.2370 grms. of silver, which would make the precipitate contain 0.1785 grm. of silver and 0.0683 grm. of selenium, the calculated amounts for 0.2468 grm. of silver selenide being 0.0666 grm. of selenium and 0.1802 grm. of silver.

The precipitate obtained in Experiment 2 was treated with a solution of gold chloride and reduced it nearly as well as does elementary selenium. The fact that both the telluride and the selenide of silver reduce gold chloride suggested an investigation of the action of the sulphide of silver on solutions of silver and gold. Silver sulphide was prepared by passing hydrogen sulphide into a solution of silver nitrate made alkaline with ammonia, the precipitate washed, dried in an air-bath, and then washed with carbon bisulphide to remove any excess of sulphur. A sample of silver sulphide prepared in this way was boiled with a solution of silver nitrate for seven days and the solution tested for sulphur without any trace being found to be present. The silver sulphide reduced gold chloride solution to metal, readily on warming but only slightly in the cold.

#### Conclusions.

Tellurium reduces a gold chloride solution completely to metallic gold either in the warm or in the cold, and the only difficulties so far as quantitative precipitation is concerned are mechanical ones. The action of tellurium on silver salts in solution is to reduce them with formation of the telluride of silver, which is still a reducing agent, as it throws out gold from solution. The behaviour of the product obtained is, in all respects, similar to that of the telluride of silver obtained by the reduction of silver tellurite and to that of the native mineral itself.

The action of selenium is similar to that of tellurium, but is not as energetic. Selenium reduces silver solutions in the cold, but has no action on gold solutions unless heated nearly to boiling, the action then being fairly rapid and complete. With silver solutions selenium forms silver selenide, which resembles silver telluride in being a reducing agent to solutions of gold salts, both of them being similar to the sulphide of silver in this respect.—*Journal of the American Chemical Society*, xxiv., No. 10.

## CONTRIBUTIONS TO OUR KNOWLEDGE OF YTTERBIUM.\*

By ASTRID CLEVE.

(Concluded from p. 302).

#### IV. ELECTROLYTIC CONDUCTIVITY.

BELOW are given the results of some experiments to determine the electrolytic conductivity of solutions of ytterbium salts, and hence the velocity of migration of the ytterbium ion.

The determinations were made according to Kohlrausch's method; thus the conductivity of salt solutions of different concentrations were ascertained by a Wheatstone's bridge and telephone.

A crystallised sulphate, which was prepared from the purest possible material of atomic weight 173.1, was used for the experiments. Series of solutions of different dilutions were made from four different preparations (I.—IV. in the following table), and each of the numbers given is the mean of several separate determinations. All values are referred to 18°. For the greatest dilutions distilled

\* From the *Zeitschrift für Anorganische Chemie*, xxxii.

water with a conductivity of at the most  $2.5 \cdot 10^{-6}$  was used in platinum vessels, and the results given have been corrected for the conductivity of the water.

$v$  denotes the volume in litres in which a grm.-equivalent equals 64.8679, of crystallised ytterbium sulphate is contained:  $\lambda$ , the equivalent conductivity at  $18^\circ$ .

| v.     | $\lambda$ . Series— |      |       |      | Mean value. | Difference. |
|--------|---------------------|------|-------|------|-------------|-------------|
|        | I.                  | II.  | III.  | IV.  |             |             |
| 3'33   | 20'9                | 20'9 | 20'7  | 20'4 | 20'7        | 14'6        |
| 6'67   | —                   | 24'7 | —     | —    | 24'7        |             |
| 33'33  | 35'0                | 35'4 | —     | 35'4 | 35'3        | 26'3        |
| 333'3  | —                   | 61'7 | —     | 61'5 | 61'6        |             |
| 666'7  | —                   | 74'— | —     | —    | 74'—        | 36'4        |
| 3333'  | —                   | —    | 97'—  | 98'8 | 98'—        |             |
| 16667' | —                   | —    | 105'— | —    | 105'—       |             |

The table shows that ytterbium sulphate in dilute solution is partially hydrolysed. Some experiments with litmus also led to the same conclusion; in concentrated solutions of the salt the litmus was pure violet, but on increasing the dilution of the same solutions with pure water the litmus became distinctly reddish.

Unfortunately, so far as I know, the conductivity of yttrium sulphate has not been determined, and thus a direct decisive comparison of the relative basicity of the yttrium and ytterbium hydroxides cannot be made. As regards the lanthanum (Muthmann, *Ber. Deutsch. Chem. Ges.*, cxxi., 1829) and praseodymium (Jones and Reece, *Amer. Chem. Journ.*, xx., 606) salts, certain information may be obtained, but this does not admit of a direct comparison, for the temperature of the research with the first salt was  $+25^\circ$ , and for the latter no temperature was stated.

#### V. SUMMARY.

In all the preceding reactions ytterbium has shown itself to be a purely trivalent element. It forms many compounds specially characteristic of the trivalent metals; of these the following examples may be mentioned:—The gold double chloride, the acid selenite, the potassium double ferrocyanide, the potassium double chromate, the periodate,  $\text{YbIO}_3$ , and the basic carbonate,  $\text{ROHCO}_3$ . Even in acid solutions neutral ortho-salts of tri-basic acid, as, for example, the phosphoric acids, are formed. Finally, mention must be made of the existence of organic salts of the type of the acid selenite, such as the acid malonate and acid tartrate, formed from two molecules of a dibasic acid by substitution of three-quarters of the acid hydrogen by the metal.

The close relationship of ytterbium to yttrium is specially apparent in the platinum cyanide. This salt is externally completely similar to the corresponding yttrium compound. There is certainly a discrepancy in the amount of water it contains, which was found to be 18 molecules, while, according to information furnished by Cleve, the yttrium and erbium platinum cyanides crystallise with 21 molecules of water. However, it would be best to make further experiments to prove whether a difference in this respect actually exists. This now appears somewhat improbable, since Benedicks has proved that gadolinium platinum cyanide, containing 18 molecules of water, has both the appearance and the crystalline form of yttrium platinum cyanide.

Ytterbium can thus be classed together with yttrium, erbium, gadolinium, and possibly other metals, which have as yet not been prepared in the pure state, in a natural group, based on the similarity of the platinum cyanides. As far as we can judge at present, ytterbium is most closely allied to erbium among these metals, both as regards the composition of many salts, and the comparatively great solubility of the sulphate.

Brauner ("Contribution to the Chemistry of Thorium," *Trans. Chem. Soc.*, 1898, p. 972) has expressed the opinion that the solubility of the oxalates of rare earths in neutral ammonium oxalate is due to the formation of complex salts, and hence may be taken as an indication of the

relative basicity of the earths, so that a soluble double oxalate is formed in greater quantity the more feebly positive the earth is.

If this is the case, ytterbium is one of the least positive of the metals of the rare earths, and takes its place between thorium and yttrium, i.e., far below praseodymium.

But then it seems strange that both gadolinium and ytterbium form a basic carbonate,  $\text{R.OH}:\text{CO}_3$ , under conditions which, in the case of yttrium, lead to the formation of the neutral carbonate. According to Brauner's arrangement, gadolinium, which is placed between praseodymium and neodymium, comes long before yttrium, and still more ytterbium, and is thus, from the interpretation which has just been put on these facts, much more strongly positive than these last metals.

On the other hand, evidence can be adduced which points to a comparatively feebly positive character in the case of ytterbium.

We must also take into consideration the decomposition of the nitrate, which begins very readily on heating, and upon which Marignac's method of separation is based. The chloroplatinate has, moreover, the composition  $2\text{YbCl}_3 \cdot \text{PtCl}_4 + n\text{H}_2\text{O}$ , which differs from the usual type for other rare earths,  $\text{RCl}_3 \cdot \text{PtCl}_4 + n\text{H}_2\text{O}$ , while the yttrium compound differs in exactly the opposite direction. Finally, must be mentioned the formation of oxy-salts, such as  $(\text{YbO})_2\text{WO}_4$  and the comparative indifference of the ytterbium earth towards acids.

### ARSENIC IN GLYCERIN.

By J. BOUGAULT.

ALTHOUGH glycerin is intended generally for external application only, a certain proportion is taken internally, often in large doses, either by itself or in various preparations; the presence of arsenic, therefore, should not be tolerated.

Commercially, a considerable quantity of glycerin is used in manipulating wine, and in this connection I am informed the users are on their guard, and every sample of glycerin used is examined carefully and especially for this dangerous impurity.

I myself have had occasion to examine glycerins destined for pharmaceutical purposes, which contained quantities of arsenic corresponding to 3 or even 5 centigrms. of arsenious anhydride per litre. And what gives importance to this observation is the fact that the examination was not of one small sample only, but of several samples taken each time from deliveries of several thousand kilograms.

Pharmacists are liable, therefore, to receive such a class of glycerin, and it is well that they should be warned of this eventuality.

The presence of the arsenic is easily detected without having recourse to the destruction of the organic matter, by precipitation in the form of sulphide. But sulphuretted hydrogen is not a convenient reagent to handle, so I recommend the following method, which is of sufficient sensitiveness, and of which the preparation and use is simple. This reagent is based on hypophosphorous acid, which was prepared some years ago as a delicate test for arsenic by M.M. Engel and Bernard (*Comptes Rendus*, 1896, vol. cxxii., p. 390; *Journ. de Pharm. et Chim.*, 1896, [6], iii., p. 413).

Twenty grms. of hypophosphite of sodium are dissolved in 20 c.c. of water, and 200 c.c. of pure hydrochloric acid added of density = 1.17. Chloride of sodium is precipitated; this is filtered off through a plug of cotton-wool.

Five c.c. of the glycerin under examination and 10 c.c. of the reagent are placed in a test-tube; after well mixing this is placed in a boiling hot water-bath. With 0.1 milligram of arsenious anhydride we obtain a brown colouration followed rapidly by a brownish flocculent



precipitate; with 0.02 milligram we still get a distinct brown colouration, which is more easily seen if compared with a similar tube not containing arsenic. Even 0.01 milligram gives a colouration, and after standing for some days a precipitate is visible at the bottom of the tube; but considering only the 0.02 milligram test, it is obvious that we can rapidly detect 4 mgrms. of arsenious anhydride per litre.

The appearance of the precipitated arsenic is very characteristic and a practised eye cannot be deceived; if, however, any doubt did exist, the precipitate could be collected and the presence of arsenic confirmed by the well-known method in the form of arseniate of silver.

It would be also very interesting to know how this arsenic comes to be in the glycerin, but the particulars in my possession do not enable me to solve the question in a very definite manner.

Glycerins for pharmaceutical use are obtained by the distillation of raw products from two different sources:—

1. Glycerins from saponification, which are produced by the saponification of fatty matters by means of lime, under pressure.

2. Glycerins, called lyes, which are residues from the manufacture of soaps. The preparation of the first kind gives no opportunity for the introduction of arsenic. As for those of the second kind, being very alkaline, they have to be saturated, before distillation, with commercial hydrochloric acid, which is always arsenical; it may be that this acid is the source of the arsenic of which a portion would be carried over mechanically during the distillation.

Distilled glycerins are then decolourised with animal black, and then often treated with a small quantity of some product to hide their more or less yellow appearance. I am not able to say with certainty whether these two operations are likely to introduce arsenic into the final product.—*Journ. de Pharm. et Chim.*, Series 6, vol. xv., No. 11.

## A METHOD FOR THE PREPARATION OF THE DIPHENYLAMINES.

By RAYMOND VIDAL.

THE formation of simple diphenylamine by heating aniline with its hydrochlorate, in a closed vessel, is effected at a relatively high temperature, and the return is limited.

The same does not hold good in the preparation of the more complex diphenylamines. In fact, it is extremely easy to effect the formation either of dioxydiphenylamine by heating the hydrochlorate of *p*-amidophenol with *p*-amidophenol, or that of amidoxydiphenylamine by heating the hydrochlorate of *p*-amidophenol with *p*-phenylenediamine, or again, that of diamidodiphenylamine by heating the hydrochlorate of *p*-phenylenediamine with this base.

In the second case the hydrochlorate of *p*-amidophenol may be replaced by the hydrochlorate of *p*-phenylenediamine, and the *p*-phenylenediamine by *p*-amidophenol.

It is advisable to carry out these reactions in closed vessels in an oil-bath at a temperature of about 200° for four hours.

The reaction is particularly sharp in the presence of a certain amount of water, about three or four times the weight of the compounds used in the operation; these latter should be used in molecular proportion.

The returns are nearly theoretical. On opening the sealed tube a white crystalline mass is discovered, rapidly turning black in the air, and accompanied by a small quantity of liquid, which may be separated by

draining. It is difficult, even when drying *in vacuo*, to obtain white diphenylamines.

The hydrochlorate of diamidophenol, when heated under the above conditions with *p*-amidophenol or *p*-phenylenediamine, forms trisubstituted diphenylamines, but these latter, more easily changed than the preceding ones, are in the form of a blackish mass, even on the opening of the sealed tube.

Monosubstituted diphenylamines are also obtained by the same method, by heating the hydrochlorates of the aromatic amines, oxyamines, or diamines with the aromatic amines, oxyamines, or diamines themselves.

In fact, the reaction discovered by Girard, limited up to the present to the formation of the most simple diphenylamine, takes place more easily with the polysubstituted aromatic amines, and the hydrochlorates of the polysubstituted amines.

This reaction, which gives only mediocre returns, when carried out in open vessels and in the dry state, gives a much larger product in a sealed vessel and in the presence of water. It may be generalised by saying that it occurs, whatever the nature or the position of the functions in the compounds used, and it is also as easy to effect as the process which consists of heating dinitrochlorobenzol-1.2.4 with the polysubstituted aromatic amines.—*Moniteur Scientifique*, Series 4, vol. xvi., Dec., 1902.

## THE CONSERVATION OF MATTER.

The balance inculcates thrift and morality generally, and weighing should be so constantly resorted to that it becomes an absolute habit. If Rudyard Kipling could but be persuaded to write a song with the refrain, "Weigh, Weigh, Weigh," which could be hummed by girls during their lessons in practical work in science, as well as sung on State occasions, he would be doing education a great service.—*Prof. Armstrong*.

As Mr. Kipling has not responded to the call, we venture to put forward the following poem, but well-meant, substitute.

WHEN you've used up all the borax, and the beads no longer charm,

When you've made sufficient sulphuretted stench,  
Will you kindly drop a rider on the graduated arm  
Of the little becker balance on the bench.

You have learnt a lot of symbols, long equations and the rest,

And of these just like a parrot you can chatter,  
But have you thought of trying on your own account to test

The Indestructibility of Matter?

Gramme weight—drachm weight—weight of a hundred grains.

(Fifty thousand boys and girls, it's all the same to-day.  
Each of them doing his own research, each of them using his brains).

Put the weights in the balance pan and weigh—weigh—weigh!

Oh, argon you have read about, and modestly you own

That you think you're fully able to declare,  
With an accuracy limited by paper-space alone,  
The percentage composition of the air.

You have seen the rose and jessamine grow climbing  
'neath the eaves,

And in autumn heard their leaves fall pitter-patter;  
But do you see you're proving, as you burn the faded leaves,

The Indestructibility of Matter?

Red leaf—dead leaf—leaf that is charred and hot,

Leaf of a rose or buttercup,—it's all the same to-day.  
Each of them serving to prove the law.—And what's to be done with the shot?

Put it into the balance pan and weigh—weigh—weigh!

\* The reagent mentioned above can be used for the detection of arsenic in a large number of pharmaceutical preparations, among which the following are the most prominent:—Phosphate of soda, the various phosphates of lime, hydrochloric and sulphuric acids, &c.

Of your knowledge of your bone and blood's components  
you are proud.

(No doubt you have a list of them by heart).

You can tell in learned language that would mystify the  
crowd

How the oxyhæmoglobin plays its part.

But you've seen the grasses growing by the margin of the  
lake,

And you've watched the browsing herd grow slowly  
fatter,

Yet have you thought you're proving, when you're lunching  
off a steak,

The Indestructibility of Matter?

Gramme weight—drachm weight—weight of a hundred  
grains.

(Fifty thousand boys and girls, it's all the same to-day.  
Each of them doing his own research, each of them using  
his brains).

Put the weights in the balance pan and weigh—weigh  
—weigh!

We have burrowed in the coal pits, we have quarried out  
the stone,

We have forged a house of iron in the flame.

But the whirl-wind and the rain-cloud they shall reap  
where we have sown,

Nought shall last of that we builded but the name.

Yet although our work shall perish, though it crumble into  
dust,

Which the four winds of the heavens freely scatter,

There is evidence convincing in that scattered iron rust  
Of the Indestructibility of Matter.

Brown rust—town rust—rust from the country gates,

Rust from an old torpedo boat, it's all the same to-day.

All of it serving to prove the law.—And what's to be done  
with the weights?

Put them into the balance pan and weigh—weigh—  
weigh!

M.S., *Central Gazette*.

## PROCEEDINGS OF SOCIETIES.

### PHYSICAL SOCIETY.

*Ordinary Meeting, December 12th, 1902.*

Mr. S. LUPTON, Vice-President, in the Chair.

MR. S. W. J. SMITH exhibited and described a Portable  
Capillary Electrometer.

This instrument is a modification of the form of capillary electrometer which consists of two wide tubes joined by a cylindrical capillary tube which may be horizontal or inclined. The apparatus contains mercury and sulphuric acid of about maximum conductivity suitably distributed in the tubes. A spring key is commonly employed with such an instrument, and keeps the two platinum terminals at the same potential unless the spring is depressed. When the spring is depressed, the terminals are put in contact with the two points, the potentials of which are to be tested for equality. In the instrument shown by the author the two wide tubes are sealed at the top to prevent evaporation of the sulphuric acid. The wide tubes are also joined near the top by another tube which allows free motion of the liquids within the apparatus. With this arrangement the instrument is made air-tight, and can, if desired, be made air-free by exhaustion of the apparatus before sealing. The distribution of the mercury can be altered most easily by means of a third cross-piece provided with a tap. To prevent the platinum wires forming the electrodes being wetted by the acid, if the apparatus should accidentally, or during transit, be laid on

its side or turned upside down, the lower ends of the tubes may be slightly constricted above the ends of the platinum terminals. If the terminals consist of amalgamated platinum-foil the constriction is unnecessary. If a spring key is made of brass the contacts frequently become unsatisfactory through surface tarnishing, and if to avoid this the bearing surfaces are made of platinum, the key may be subject to thermo-electric effects. Such a key cannot conveniently be fastened on the same stand as the rest of the instrument, for, unless the stand and the support on which it rests are very rigid, the pressure necessary to depress the spring produces sufficient movement of the meniscus, during the act of depression, to render the detection of minute changes of surface-tension impossible. These difficulties are overcome by using a key consisting of a U-tube closed at one end, communicating at the other with a pneumatic pressure-ball, and containing mercury in the bend. Three platinum wires are fused into the tube, so that by squeezing the ball the same change of contacts is produced as by pressing the lever of an ordinary spring key. This form of key can be extended so that a slight pressure applied to the ball makes it work as an ordinary electrometer-key, and greater pressure reverses the contacts between the terminals and the points under investigation. With such an arrangement the sensitiveness of the instrument is doubled, and by using a microscope magnifying 50 diameters a potential difference of  $\frac{1}{1000000}$  volt can be detected without difficulty. The instrument, used as a surface-tension galvanometer, is more convenient than an ordinary galvanometer with a magnetic system because there is no suspension, no lamp and scale, and practically no levelling.

Dr. R. T. GLAZEBROOK congratulated the author, and said that workers with the capillary electrometer would appreciate the improvements introduced. He said that the author had given the minimum E.M.F. which the instrument could detect, and asked what was the maximum E.M.F. which could be safely applied. He referred to the advantages of the mercury keys exhibited, and remarked that, by their use, many difficulties were obviated.

Dr. LEHFELDT said the mercury keys were improvements on those used by Professor Onnes. In these keys the contacts were changed by the motion of mercury brought about by tilting. The tubes containing the mercury were exhausted to prevent oxidation.

Mr. SMITH, in reply to Dr. Glazebrook, said that the maximum E.M.F. should not exceed half a volt.

A paper "On Astigmatic Aberration" was read by Mr. R. J. SOWTER.

This paper affords a simple explanation of some of the shadow phenomena observed by Prof. S. P. Thompson in his experimental researches on the aberration of lenses, namely, in those experiments in which the aberration is wholly or in part astigmatic. It is shown that the presence of astigmatic aberration in a non-homocentric pencil is accountable merely for the twist or rotation produced in the shadow or image formed by an object placed in, or moved about in, the pencil. The paper commences with a determination of the equation of a surface bounded by the rays of an astigmatic pencil. The focal lines are taken at right angles to each other, and it is assumed that the bounding surface is generated by an ellipse which moves from the primary to the secondary focal line parallel to itself, and to a plane which is at right angles to a line perpendicular to the two focal lines. The equation to the surface is first obtained in Cartesian co-ordinates and then transformed into an equation containing two variables; one the distance of the cutting plane from the primary focal line, and the other the eccentric angle of a point on the elliptic section obtained by the cutting plane. The author then proceeds to determine the rays or generators of this skew surface, and shows that the general equation of a ray can be expressed by the simple formula  $\phi = \text{constant}$ , where  $\phi$  is the eccentric angle of any point on the ray. The simplicity of this equation points to an easy

method of constructing a string model of the astigmatic surface. The paper then deals with the shadows produced by an object placed in an astigmatic pencil. A straight wire placed at an angle across the beam and cutting the axis will depict upon a screen placed beyond the secondary focal line a straight-line shadow at a definite inclination. If such a wire is moved, without varying the inclination, from the primary to the secondary focal line, then the shadow twists through  $90^\circ$ . If in an experimental pencil or beam there are astigmatic and spherical aberrations present, then a straight wire placed across the beam and moved about would not cast a straight-line shadow but would cast a distorted shadow, such as a figure S, which would be displaced through an angle, the displacement being due to the astigmatic aberration present in the beam.

Prof. EVERETT asked if the paper dealt with a zone or with the whole of a pencil. He said that generally the focal lines were not at right angle. In the ordinary case of a lens at  $45^\circ$  to the axis of a pencil, the secondary focal region was very narrow, and it was possible to trace a curved line through it and call it the secondary focal line, but this line was not at right angles to the axis of the pencil. Most sections in the primary focal region were closed curves cutting themselves twice.

Dr. R. T. GLAZEBROOK said that much of the author's work was based on the assumption that the focal lines were at right angles. The analysis had been simplified by assuming that the surface is formed by the motion of an ellipse, but he thought this assumption ought to be justified. He showed how the results arrived at in the paper could be obtained more generally without any such assumption.

Mr. PRICE referred to a model similar to the elliptical trammels, but with the grooves in different planes. He said that if two fixed points in a bar were constrained to move along these grooves, then any other point on the bar traced out a surface which had characteristics resembling those of an astigmatic surface.

The AUTHOR, in reply, said that he had made certain assumptions to give a simple explanation of the shadow phenomena observed by Prof. Thompson. The fact that a generator of the surface could be expressed by the equation  $\phi = \text{constant}$  rendered this explanation easy. With reference to Prof. Everett's question, he said that the analysis applied to the bounding surface of an astigmatic beam.

Prof. L. R. WILBERFORCE exhibited apparatus for a Lecture Experiment on Gaseous Diffusion.

In Graham's experiments on diffusion through porous septa, the gas experimented upon was contained in a vessel inverted over water, and the pressure was kept approximately atmospheric by applying a counterpoise to the vessel. This adjustment, however, is imperfect owing to the weight of the water displaced by the material of the vessel. Prof. Wilberforce showed that, by suspending the vessel from one arm of a balance rendered suitably unstable by a weight above the central knife-edge, a compensating effect could be introduced, and the pressure kept sensibly constant for a considerable range of motion of the vessel. He pointed out that this device could also be utilised for the measurement of pressure.

A paper on "Vapour-density Determinations" by Prof. Sir W. RAMSAY and Dr. STEELE was read by Sir W. Ramsay.

The accurate determination of the densities of gases has been for long an object to which chemists have paid attention. On the other hand, the densities of vapours has only been roughly estimated, as a means of arriving at a conclusion regarding molecular weights; whilst accurate molecular weights have been deduced from the results of analysis, and from previous determinations of atomic weights. The present paper gives a detailed account of some accurate experiments on the densities of vapours over a large range of pressure carried out by a modification of Gay-Lussac's method. This method has

the advantage that while densities are being determined, compressibilities can, within certain limits, be simultaneously estimated with the same sample of material. Berthelot has shown how a knowledge of the compressibility and density of the more permanent gases can be used to compare their relative weights when, if Avogadro's hypothesis be granted, equal volumes contain equal numbers of molecules. This can be done by assuming the compressibility to be a linear function of the pressure, and calculating the value of  $p_v$  at zero pressure. The ratio of the  $p_v$ 's will then be the ratio of the densities when equal volumes contain equal numbers of molecules. This method has been applied graphically to the results of the authors' experiments on vapours by plotting as ordinates the pressures, and as abscissæ the values of  $p_v/T$ . Where the curve cuts the line of zero pressure the theoretical value of  $p_v/T$  has been reached. The evidence goes to show that the densities of certain compounds calculated for zero pressure are not proportional to their molecular weights deduced from the atomic weights of the elements which they contain. This conclusion involves one, or it may be several, of a series of assumptions enumerated in the paper. These assumptions are fully investigated and discussed, and the authors suggest that it may be possible that the atomic weights of the elements depend on the proportion in which they are present in the compounds which contain them.

Prof. EVERETT referred to the work of Regnault and Amagat upon the variation of the product  $p_v$  at high pressures.

Dr. A. GRIFFITHS, in commenting on the theoretical basis of the comparison of densities, indicated his lack of confidence in one of the fundamental hypotheses, viz., that the temperature of a given gas depends simply on a single factor, the mean square of the linear velocity of the molecules.

Dr. LEHFELDT asked what standard substance had been employed throughout the investigation, and whether the authors were satisfied that the different graphs of  $p_v/T$  all cut the zero-pressure line in the same point. The experiments seemed to be in agreement with Lord Rayleigh's statement that at low pressures  $p_v/T$  is a constant for the permanent gases.

Sir W. RAMSAY said that the standard substance used was oxygen, and that the curves obtained for any vapour at different temperatures practically cut the zero-pressure line at the same point.

The Society then adjourned until January 23rd, 1903.

## NOTICES OF BOOKS.

*The New Volumes of the Encyclopædia Britannica.* The Eighth of the New Volumes, being Vol. XXXII. of the Complete Work. London: *The Times*. Edinburgh: Adam and Charles Black. 1902. Pp. 872.

THE eighth number of the new volumes comprises those subjects coming within the alphabetical headings P R I (Pribiloff Islands) and S T O (Stowmarket). Though not so rich in items of scientific interest as some of its predecessors, there are still a fair number of articles which call for special notice.

In the first place there is the Prefatory Essay by Karl Pearson, entitled "The Function of Science in the Modern State"; this article is of considerable length and deals with a very complex problem from various points of view. "The future," says the author, "is to the nations which not only realise the international struggle in all fields of activity, but consciously develop all the factors of national efficiency with this end in view. . . . Brute force, strength and bravery, material wealth, have in turn been dominant in the State; to-morrow will be marked by the dominance of intelligence."

The future will be an era of specialism, in which every

one will have to be carefully trained in some particular art, or else not only the individual, but the State, will have to drop out of the struggle of nations. To this end science is becoming more and more the paramount factor, and the proper recognition of science by the State is the most encouraging sign of modern times. The author advocates the formation of a "State Science Counsel," with a patent like that of the existing King's Counsel, as a spur or incentive to young men, and a reward to those who have distinguished themselves; we may point out that the "Order of Merit" recently founded seems to fulfil this requirement to some extent, though the standard has been fixed too high for the "general practitioner" of science.

The article on "Public Health," by A. Wood Renton, is short, but sets forth clearly some of the requirements and provisions of the laws on this subject. "The Theory of Radiation," by Dr. J. Larmor, Sec. R.S., covers nearly eight pages, and, as its title indicates, is essentially of a mathematical character; it deals with the law of distribution of energy, pressure of natural radiation, the temperature of an isolated ray, the temperature of the sun, &c.

"Sewage Disposal," by the late Santo Crimp, M.Inst.C.E., is an article on an important practical subject which is claiming more and more attention every year. The author's principal concern is how to deal with sewage at the outfall so as to render it innocuous; but to do this we must have a clear idea of its composition and the circumstances of its delivery. The best way of disposing of sewage is to let it run direct into the sea, under certain restrictions,—that is if we do not wish to save any of the valuable nitrogenous matter; the sea purifies it rapidly by means of the living organisms with which it (the sea) abounds; but this method of disposal is not always practicable, and recourse must be had to other means, such as precipitation, or the use of bacteria beds and septic tanks. If the beds are properly aerated and of sufficient area, the aerobic organisms establish themselves in prodigious numbers and attack the organic matter, breaking it down into harmless soluble and gaseous products with great rapidity.

"Silver" is dealt with by H. O. Hofman, Ph.D. He describes the various means adopted for extracting silver from its ores, such as amalgamation, including the old "Patio" process, and the more modern though still old "Washoe" process. Roasting, lixiviation, and the different forms and combinations of these methods are also described. The article is on the metallurgy of silver only, and does not touch assaying.

Prof. G. E. Hale, of the Yerkes Observatory, contributes a valuable article on "Spectroscopy," covering ten pages, in which solar and stellar spectroscopy naturally take a prominent place. There are many other articles of great general interest, besides those appealing to individuals devoted to special subjects.

*The Year-book of New South Wales.* Compiled by the Editor of the "Year-book of Australia," for Circulation by the Agent-General in London. 1903. Pp. 168.

INTENDING emigrants, or even travellers, to Australia will find a good deal of both valuable and interesting information in this "year-book"; it contains matter of all kinds—political, social, commercial, historical, geological, &c.; in fact, there is a little of everything.

We have referred on a former occasion to the diamond industry of New South Wales, and are glad to notice that an increase in the output is expected for the year 1902; the Inverell Diamond Fields Company commenced washing on a large scale about a year ago, and their results will doubtless cause a large increase in the quantity of stones found.

The educational system of this State is established by Acts of the local Parliament dealing with primary and secondary schools and with the University; it is interesting to note that though the number of schools and

the number of pupils enrolled and attending increase in a regular manner, the expense, on the other hand, keeps about the same, or at any rate is not increasing anything like so rapidly as the number of scholars.

*Twenty-fifth Annual Report of the Connecticut Agricultural Experiment Station for the Year Ending October 31st, 1901.* New Haven, Conn.: The Tuttle, Morehouse, and Taylor Company. 1902. Pp. 446.

THE amount of work done at this experiment station is large and varied; it includes not only the examination of commercial fertilisers, of which 429 samples were analysed, but also that of food products, botanical, horticultural, and entomological work, the improvement of waste lands, &c.

The section on fertilisers covers a wide field, and the review of fertilisers gives some valuable information; the value of fertilisers must naturally vary with the soil, crop, and weather, and widely different results may be expected from different places; as a broad general rule it is true that ground bone, superphosphates, fish scraps, dried blood, potash salts, &c., have a high agricultural value, which is related to their trade value, and to some extent determines the latter; but the rule has many exceptions, and in some instances the two do not correspond at all.

An important investigation is being carried out by Dr. Osborne and Mr. I. F. Harris on the constitution and properties of nucleic acid of the wheat embryo, which promises to give very valuable results; the conclusions they have arrived at so far are given in fourteen paragraphs, which are too lengthy, however, for more than a passing reference.

## CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

*Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences.* Vol. cxxv., No. 22, December 1, 1902.

**The Temperature of Ignition and Combustion of the Three Varieties of Carbon in Oxygen.**—Henri Moissan.—The author performs a series of experiments, and finds that in every case the combustion of the different varieties of carbon takes place at temperatures which are higher as the polymerisation of the carbon increases. Diamonds become incandescent in oxygen at 800–875°; graphite, at about 650–700°; and amorphous carbon, between 300° and 500°. But each of these rapid reactions is preceded by a reaction becoming slower as the temperature is lowered from the ignition-point.

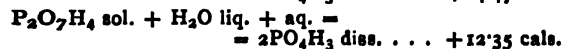
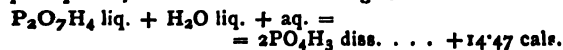
**Composition of Gaseous Hydrates.**—M. de Forcrand.—The author, by means of the general relation  $Q/T=30$ , determines the composition of certain gaseous hydrates, which composition would be impossible to determine by direct analysis. He first applies his rule to hydrate of chlorine, and finds  $Cl_2+7H_2O$  for its composition; this being verified by direct analysis. In most other cases the first method only is applicable, because the total heat of solidification of the gaseous molecule is not known. Results are obtained for the hydrates of nitrogen, marsh-gas, carbon dioxide, nitrous oxide, ethylene, acetylene, phosphoretted hydrogen, sulphuretted hydrogen, chlorine, bromine, and several other gases.

**Transformation of Pyrophosphoric Acid into Orthophosphoric Acid.**—H. Giran.—When pure crystalline pyrophosphoric acid is prepared from the syrupy acid having the theoretical composition  $P_2O_7H_4$ , and this is kept for some time at a temperature of  $-10^\circ$ , the acid crystallises out in little white grains of spheroidal form.

The author measures directly the heat of solution of this crystalline acid :—



He then finds the heat evolved during the transformation of solid pyrophosphoric acid (liquid or dissolved) to orthophosphoric, and obtains the following results :—



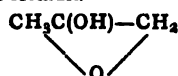
The difference in these two numbers gives the heat of fusion of the pyrophosphoric acid.

**Manganese Aluminate.**—Em. Dufau.—By means of Moissan's electric furnace alumina and oxide of manganese can easily be made to unite. The author then makes a detailed study of the product,  $Al_2O_3.Mn$ .

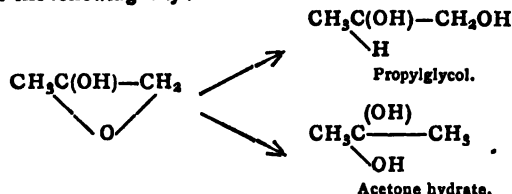
**Estimation of Manganese.**—H. Baubigny.—It has been previously found that the salts of manganese protoxide can be transformed into those of the insoluble peroxide by the action of certain reagents. The author's investigations on these reactions show that the oxidation of manganese salts by the persulphates takes place only in a very strongly acid medium.

**Action of Chlorine and Bromine on the Mononitrated Veratrols.**—H. Cousin.—The author determines the constitutional formulæ of a certain number of tri-substituted derivatives of pyrocatechin or its methylic ethers. He also describes two new bodies—a dichlor-3-nitrated veratrol and a dichlor-4-nitrated veratrol.

**Hydrogenation of Acetol.**—A. Kling.—Free acetol in alkaline, neutral, or acid solutions, whether hot or cold, exists at least partially in a state which has not the usual constitution represented by the formula  $CH_3CO.CH_2OH$ , but probably by the formula—

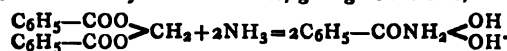


The hydrogenation of this alcohol-ether oxide takes place in the following way :—



The hydrate of acetone is transformed into acetone or its product of hydrogenation—Isopropyl alcohol.

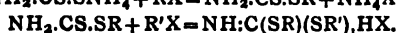
**Action of Fatty Amines on Dibenzate.**—Marcel Descudé.—The author has already shown that ammonia reacts on methylene dibenzate, giving benzamide,—



This principal reaction is accompanied by two secondary reactions, by which water and formic aldehyde are liberated. From this, ammonium benzoate and hexamethylene tetramine are formed, and no formic aldehyde in the free state. By substituting for ammonia the fatty amines, the reaction is similar; the secondary amines react much less easily than the primary amines, and the tertiary amines have no reaction.

**Action of the Halogenated Ethers on Ammonium Thiosulphocarbamate.**—Marcel Delépine.—The author studies the action of the halogenated ethers on ammonium thiosulphocarbamate,  $NH_2.CS.SNH_4$ . One molecule of these ethers produces the thiosulphocarbamic ethers or non-substituted dithiourethanes; a second produces salts of the imido-dithiocarbonic ethers, also non-substituted.

The reactions are similar to those which the author previously describes for the primary amines :—



*Bulletin de la Société Chimique de Paris.*

Series 3, Vol. xxvii., No. 9.

**The Oxides of Molybdenum.**—Marcel Guichard.—Already noticed.

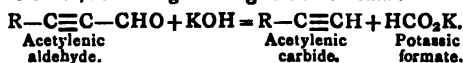
**Condensation of True Acetylenic Carbides with Formic Aldehyde; General Method for the Synthesis of Primary Alcohols containing an Acetylenic Function.**—Ch. Moreau and H. Desmots.—During the condensation of true acetylenic carbides,  $R-C\equiv CH$ , with formic aldehyde,  $CH_2O$ , the authors found a very simple method for the synthesis of primary acetylenic alcohols,  $R-C\equiv C-CH_2OH$ . Their experiments were carried out on three carbides :—Cenanthylidene (heptene-1),  $CH_3-(CH_2)_4-C\equiv CH$ ; caprylidene (octene-1),  $CH_3-(CH_2)_5-C\equiv CH$ ;

and phenylacetylene,  $C_6H_5-C\equiv CH$ . After numerous unsuccessful trials, both hot and cold, with commercial formal and carbides in the free state, in the presence of various condensation reagents (potash, hydrochloric and sulphuric acids, and chloride of zinc), they tried the effect of the solid polymer,  $(CH_2O)_n$ , known as trioxymethylene, on the soda-carbides. These experiments were quite successful. The operation consisted of putting the soda-carbide and the trioxymethylene in contact in the presence of absolute ether, and then decomposing the raw product of the reaction with water. Analysis showed that the new bodies obtained result purely and simply from the addition of the elements of the formic aldehyde,  $CH_2O$ , to those of the carbides,  $R-C\equiv CH$ . They are not precipitated by the reagents of the true acetylenic carbides,  $R-C\equiv CH$ ; for example, nitrate of silver in alcoholic solution has no action on them; thus the active hydrogen of the original true acetylenic carbides has disappeared; and from this it follows that it has been replaced by the alcoholic functional group, which, because of its terminal position, is necessarily primary. Consequently, these compounds must have for their general formula  $R-C\equiv C-CH_2OH$ , and they are primary alcohols containing an acetylenic function.

**Condensation of the True Acetylenic Carbides with the Aldehydes; General Method for the Synthesis of Secondary Alcohols with an Acetylenic Function.**—Ch. Moreau and H. Desmots.—Already noticed.

**Condensation of the Formic Ethers with the True Acetylenic Carbides.** Method for the Synthesis of the Acetylenic Aldehydes.—Ch. Moreau and R. Delange.—Already noticed.

**Decomposition of the Acetylenic Aldehydes by the Alkalis.**—Ch. Moreau and R. Delange.—Like the acetylenic acetones, the acetylenic aldehydes decompose by hydration under the influence of boiling solutions of the alkalis; there is regeneration of the acetylenic carbide, on the one hand, and production of an alkaline formate on the other, according to the general formula :—



Such, at least, is the equation which represents exactly the phenomenon which occurs with phenylpropionic aldehyde. In the decomposition of amylpropionic aldehyde,  $CH_3-(CH_2)_4-C\equiv C-CHO$ , it was found that, besides cenanthylidene, small quantities of methylamylketone, and even traces of caproic acid, were formed.

**Condensation of the True Acetylenic Carbides with the Ether Salts; Methods for the Synthesis of the Acetylenic Acetones and the  $\beta$ -Ketonic Ethers.**—Ch. Moreau and R. Delange.—Already noticed.

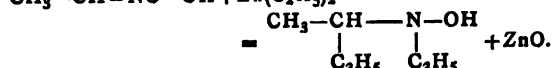
**The Extraction of Reducing Sugars (Monoses).—**C. Tanret.—The problem of determining the nature of sugars in a mixture, when both hydrolysable sugars and reducing sugars are present, would be much simplified if the mixture could be divided into two portions, one containing the hydrolysable sugars and the other the monoses. The author has attempted to do this by means of phenylhydrazine; theoretically, the monoses must be transformed into phenylhydrazones; these extracted and then decomposed to liberate the sugars. The best means of separating these hydrazones from such a complicated mixture was found to be by shaking them up with acetic ether. The extraction of glucose, levulose, invert sugar, galactose, sorbose, arabinose, &c., is described in detail; but though excellent in theory, this method is less so in practice; part of the sugars is converted into osazones, and another part escapes the reaction altogether, but in spite of all difficulties this method with phenylhydrazine allows from 77.0 per cent (levulose) to 92.0 per cent (glucose) of the monoses to be extracted from a mixture containing them.

**Some Iodised Phenols.**—P. Brenans.—Already noticed.

**Campholenic and Nitro-campholenic Acids; their Constitution.**—A. Behal.—A long paper not suitable for abstraction.

## MISCELLANEOUS.

**Action of Peroxide of Hydrogen on the Fatty Amines.**—L. Mamlock and R. Wolfenstein.—Two oxides of triethylamine,  $(C_2H_5)_3NO$ , have been described; one of them distilling without decomposition is obtained by means of zinc-ethyl and nitroethane; the other is non-volatile, and is obtained by means of hydroxylamine and iodide of ethyl (Dunstan, Golding, *Bull. Soc. Chim.*, vol. xxiv., p. 238; Bewad, Lechman, *Ibid.*, vol. xxiv., p. 792). The authors show that the product of the first reaction is a secondary hydroxylamine, which by reduction gives the secondary ethylbutylamine,  $CH_3-CH_2-CH_2-CH_2-NH-CH_2-CH_3$ , and which is formed according to the following equation:—



Analogous experiments have been carried out on the propyl series; oxides of tripropylamine (prepared from hydroxylamine or dipropylhydroxylamine, propylate of soda, and iodide of propyl at 65° for two days, or again from tripropylamine and peroxide of hydrogen in cold acetic solution) only exists in the form of the hydrate,  $(C_3H_7)_3N(OH)_2$ , a crystalline mass which re-forms tripropylamine under the influence of  $PCl_5$ ,  $NHO_2$  or  $SO_2$  in boiling aqueous solution; the picrate fuses at 130°. Sulphurous anhydride unites with this hydrate at 10°, giving anhydrous oxytripropylsulphamic acid,  $(C_3H_7)_3NSO_3$  (a general reaction of tertiary hydroxylamines); it is in the form of silky crystals, fusible at 150°, soluble in the organic solvents with the exception of ligroin, insoluble in water, which, when heated, transforms them into sulphate of tripropylamine; this anhydride is also obtained by passing  $SO_3$  gas through tripropylamine at 0°. Dipropylamine, treated first in the cold and then hot with  $SO_2Cl_2$ , gives the chloride  $(C_3H_7)_2NSO_2Cl$ , which is transformed by boiling water into dipropylsulphamic acid,  $(C_3H_7)_2N SO_3H$ , in crystals fusing at 135°, soluble in water and benzene, insoluble in ligroin, identical with the product of the action of  $SO_2$  on dipropylhydroxylamine. Bewad's base (the secondary ethylbutylhydroxylamine) fixes  $SO_2$  in benzenic solution, giving the secondary ethylbutylsulphamic acid, a white amorphous mass, soluble in the usual solvents with the exception of ligroin, fusible at 89–93°; the barium

salt is very soluble in water.—*Berichte*, vol. xxxiv., p. 2499.

**New Method for the Preparation of Dehydromucic Acids; its Salts and Ethers.**—P. A. Yoder and B. Tollens.—*Preparation.*—Fifty grms. of mucic acid are heated for forty minutes to 133–137° with 100 grms. of  $H_2SO_4$ ; after cooling, 200 c.c. of water are added, and the whole is heated for ten minutes on the water-bath; the next day it is filtered and washed with a little cold water; the impure acid is then dissolved in 800 c.c. of boiling water, and the necessary amount of barytic hydrate added. The solution is filtered, decolourised with charcoal, and finally acidulated with hydrochloric acid, when 12 grms. of dehydromucic acid are deposited. The authors have tried several other methods for its preparation, but have found them all inferior to this one; they have observed in particular, that, contrary to the opinions of Schmidt and Cobenzl, mucate of potash does not give dehydromucic acid when heated. The following salts are described:—*Potassium*, crystalline, with 1.5 or 2  $H_2O$ ; *sodium*, with 4  $H_2O$ ; *ammonium* (anhydrous), in rhombic tables having angles 122.3–122.5°, and 56.9° to 58.6°; *strontium*, with 6  $H_2O$ ; *magnesium*, with 6  $H_2O$ ; *lead*, insoluble in water; *cadmium*, with 4 or 4.5  $H_2O$ ; *copper*, small crystals containing 2.5 or 3  $H_2O$ , and giving pyromucic acid on calcination. The ethers of dehydromucic acid were prepared by the hydrochloric acid method. Their constants are given in the following table:—

Boiling-point. Pressure. Fusing-point.

|                         | M.m.     |    |
|-------------------------|----------|----|
| Dimethylic ether ..     | 154–156° | 15 |
| Diethylic ether ..      | 167–168° | 16 |
| Di-n-propylic ether ..  | 177–178° | 15 |
| Di-isopropylic ether .. | 156–159° | 13 |
| Di-n-butyric ether ..   | 186–189° | 13 |
| Di isobutylic ether ..  | 172–174° | 13 |
| Di-isoamyl ether ..     | 207–211° | 13 |

—*Berichte*, vol. xxxiv., p. 3446.

## MEETINGS FOR THE WEEK.

SATURDAY, 27th.  
TUESDAY, 30th.  
THURSDAY, Jan. 1st.

Royal Institution, 3. (Christmas Lectures, adapted for Young People). "On Locomotion on the Earth, through the Water, in the Air," by Prof. H. S. Helle-Shaw, LL.D., F.R.S., M.Inst.C.E.

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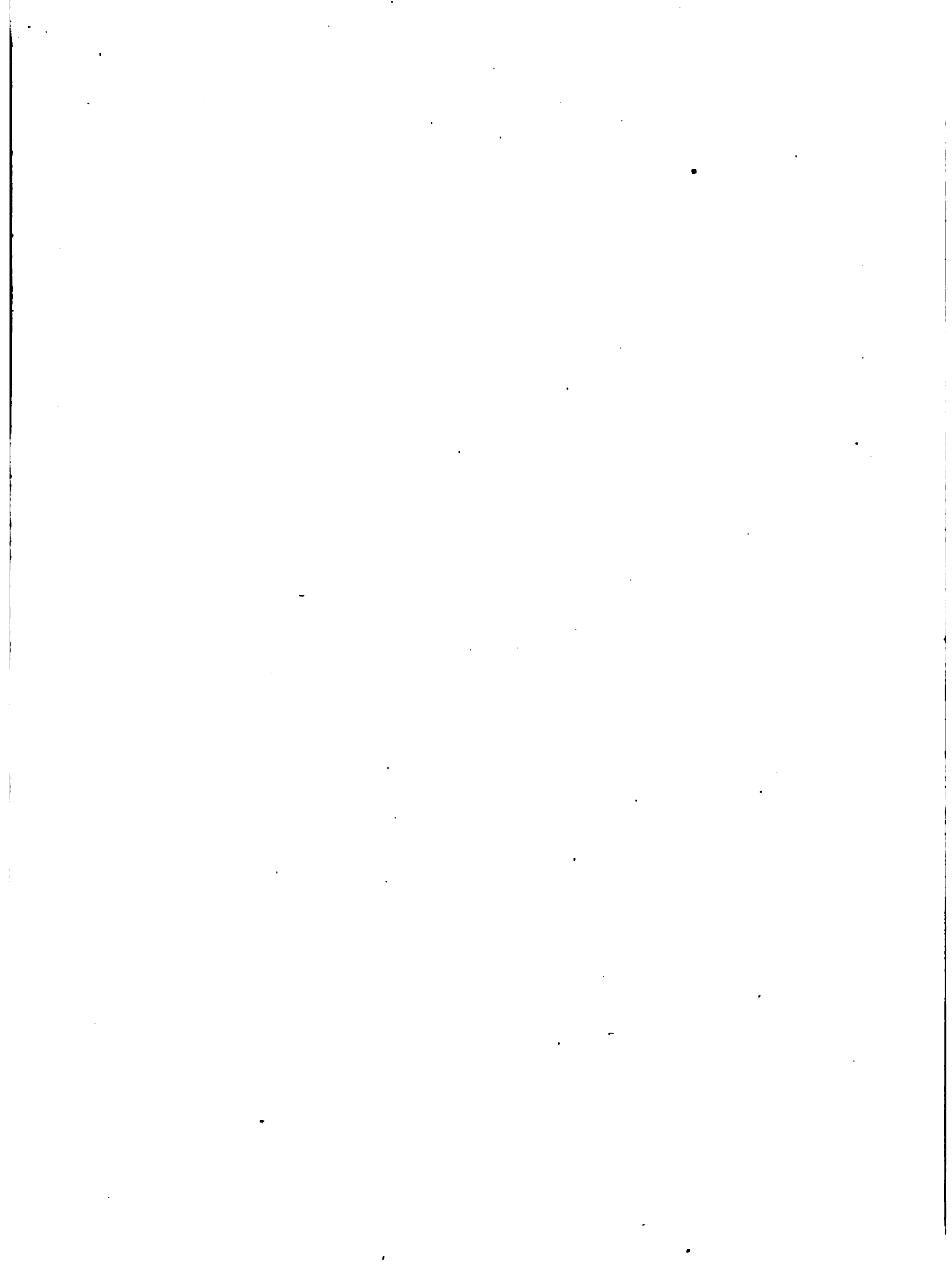


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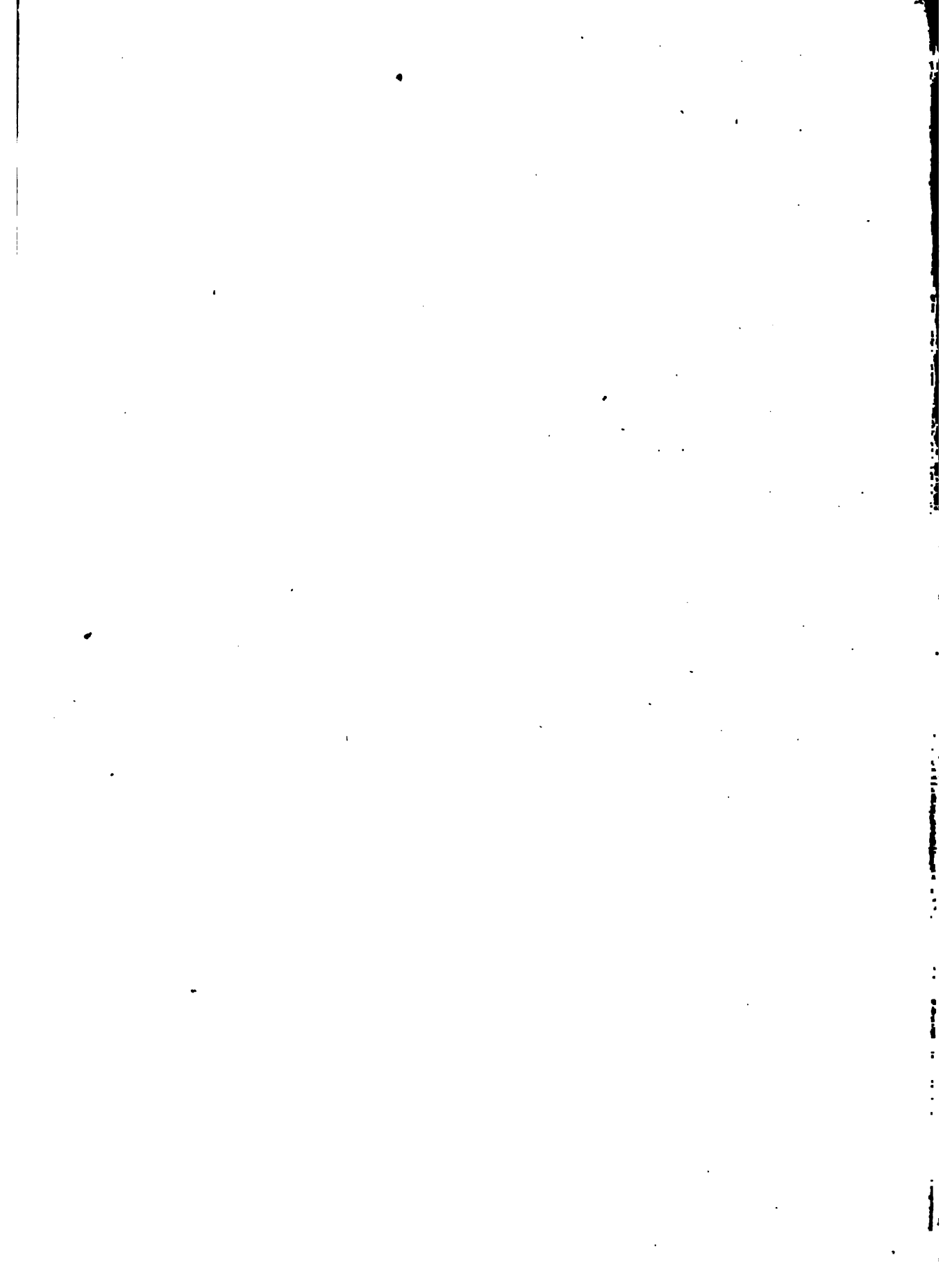














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